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Laboratory Characterization Tests for Antimisting Fuel

FAA TECHNICAL CENTER
Atlantic City International Airport
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Joseph Wilson
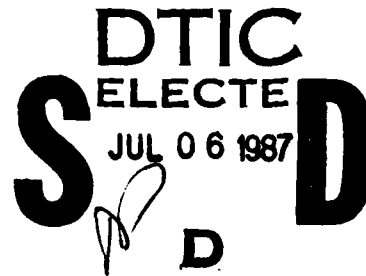
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Final Report

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16. Abstract

Experiments have shown that FM-9™ antimisting fuel has the potential for precluding the fine mist and associated fireball generation in aircraft post-crash situations while allowing for the restoration of the filtration and antomizing characteristics required for aircraft operation. The Federal Aviation Administration, the Royal Aircraft Establishment, the National Aeronautics and Space Administration, the Jet Propulsion Laboratory, Southwest Research Institute, and Pratt and Whitney Aircraft developed many specialized laboratory characterization tests throughout the antimisting fuel program to evaluate the antimisting properties, the degradability, the composition, and rheological properties of FM-9 antimisting fuel and the physicaal properties of FM-9 slurry used in the inline blending process for antimisting fuel. This report documents all the laboratory characterization tests that were successfully developed and used as a standardized test method during the program. A literature search was done, and a detailed review and analysis of each test method was conducted. This led to the following collection of information for each test method: (1) background on the intent and operation of the test method and theory behind the rneological property that the test method is evaluating relative to antimisting fuel, (2) description of the test apparatus along with a general overview of the operational procedure of the test method (3) equipment specifications for the test apparatus (4) an accurate and detailed test procedure for the test method and (5) an interpretation of the test results and a discussion of the effectiveness, repeatability, applications, and limitations of the test method.

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EXECUTIVE SUMMARY

Experiments have shown that FM-9 antimisting fuel has the potential for precluding the fine mist and associated fireball generation in aircraft post-crash situations while allowing for the restoration of the filtration and atomizing characteristics required for aircraft operation. The Federal Aviation Administration (FAA), along with others have developed many specialized laboratory characterization tests for antimisting fuel. The tests evaluate the antimisting properties, degradability, composition, and rheological properties of FM-9 antimisting fuel and the physical properties of FM-9 slurry used in the inline blending process. This report documents the laboratory characterization tests that were successfully developed and used as a standardized test method during the program. A literature search was done, and a detailed review and analysis of each test method was conducted. This led to the following for each test method:

1. Background on the intent and operation of the test method and theory behind the rheological property that the test method is evaluating relative to antimisting fuel.

2. Description of the test apparatus along with a general overview of the operational procedure of the test method.

3. Equipment specifications for the test apparatus.

4. An accurate and detailed test procedure for the test method.

5. An interpretation of the test results and a discussion of the effectiveness, repeatability, applications, and limitations of the test method.

An evaluation of the information that was collected on each of the laboratory characterization tests led to the following conclusions.

1. Laboratory characterization tests are available to adequately evaluate antimisting fuel relative to the following: (a) the antimisting properties of batch and inline blended AMK, (b) the degree of intentional degradation produced by a degrader, (c) the degree of unintentional degradation produced in an aircraft fuel system, (d) the filtration characteristics of degraded AMK, and (e) the composition and physical properties of AMK and FM-9 slurry.

2. The primary quality control tests for antimisting fuel are the Filter Ratio Test, the Cup Test, and Solids Test, and the Turbidity (Clarity) Test.

3. The only standardized laboratory characterization test that was developed as a near real-time quality control test was the Die Swell Test.

4. The laboratory characterization tests that are included in this report should provide the necessary information to assess the quality of the fuel relative to fire protection and aircraft system compatibility.

5. Attempts to make direct measurements of the normal stress and extensional viscosity properties of FM-9 antimisting fuel at high shear rates have been unsuccessful to date.

1. INTRODUCTION.

1.1 BACKGROUND.

Fire fatalities associated with impact-survivable aircraft crashes are a major concern in civil aviation and for the Federal Aviation Administration (FAA). Typically, during an impact-survivable aircraft crash, large quantities of aircraft turbine fuel are expelled from ruptured fuel tanks and fuel lines into the high velocity airstream around the aircraft. Under these conditions, the fuel that is sheared by the high velocity airstream results in a highly flammable mist which is easily ignited and at times explosive. The resulting fire then ignites pools of fuel in and around the damaged aircraft.

The Federal Aviation Administration (FAA) has been involved in research and development efforts to minimize the post-crash fire hazard of aviation turbine fuel for many years. The latest development in a series of antimisting kerosene (AMK) additives is FM-9, produced by Imperial Chemical Industries of Americas (ICI Americas). FM-9 polymer is added to Jet A aircraft turbine fuel at a nominal concentration of 0.30 percent. Experiments have shown that FM-9 AMK has indicated the potential for precluding the fine mist and associated fireball generation in aircraft post-crash situations. FM-9 AMK also exhibited the potential for allowing the restoration of the filtration and atomizing characteristics required for aircraft engine and fuel system operation.

Using the antimisting kerosene additive FM-9, the FAA, in conjunction with the Royal Aircraft Establishment (RAE), United Kingdom, completed a 2-year research and development program to determine tne feasibility of using antimisting fuel in the civil aviation aircraft fleet. Upon completion of this phase of the antimisting fuel program, the following conclusions were reached. First, the research determined that FM-9 antimisting fuel could inhibit the formation of the fine fuel mist that forms during an aircraft crash and, thus, it would reduce the post-crash fire hazard of an impact-survivable aircraft crash. Second, researchers determined that there were no problems associated with the use of AMK that could not be solved by further research using the technology available at the time (reference 1).

The successful completion of the feasibility phase of the AMK program led the FAA to the Controlled Impact Demonstration (CID) in order to demonstrate the effectiveness of FM-9 AMK in reducing the post-crash fire hazard during a full-scale aircraft crash.

The CID took place on December 1, 1984, when a Boeing 720 was remotely piloted into a prepared impact area on the desert at Edwards Air Force Base in California. The impact area contained obstacles that would normally be encountered in an airport type environment and also contained mechanical devices that would insure that the wing fuel tanks would be ruptured. The aircraft was crashed at a set sink rate and forward velocity. The impact area, sink rate, and forward velocity were determined by earlier research that defined a typical crash scenario from analysis of previous aircraft crash accident data.

The actual aircraft crash during the CID did not follow the typical crash scenario that was to have been simulated and the aircraft became involved in a post-crash fire.

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1.2 OBJECTIVE.

During the time period between 1978 when research was initiated on antimisting fuel using the newly-developed AMK additive FM-9 and 1984 when the CID took place, many laboratory characterization tests were developed to evaluate the antimisting properties of FM-9 AMK. The FAA, the RAE, the National Aeronautics and Space Administration (NASA), the Jet Propulsion Laboratory (JPL), Southwest Research Institute (SWRI), and Pratt and Whitney Aircraft (PWA) developed numerous laboratory tests to evaluate the following:

1. The antimisting properties of batch and inline blended AMK.

2. The degree of intentional degradation produced by a degrader.

3. The degree of unintentional degradation produced in an aircraft fuel system.

4. The filtration characteristics of degraded AMK.

5. The composition and physical properties of AMK and FM-9 slurry.

The principal intent of this report is to document all of the laboratory characterization tests that were used as a standardized test method during the antimisting fuel program.

Table 1.1 shows the AMK laboratory characterization tests in this report as well as the primary use of each test method. A literature search was conducted on antimsisting fuel research that contain information on the laboratory characterization tests listed in table 1.1. The review and analysis produced the following collection of information on each of the laboratory characterization tests.

1. Background on the intent and operation of the test method and theory behind the rheological property that the test method is evaluating relative to antimisting fuel.

2. Description of the test apparatus along with a general overview of the operational procedure of the test method.

3. Equipment specifications for the test apparatus.

4. An accurate and detailed test procedure for the test method.

5. An interpretation of the test results and a discussion of the effectiveness, repeatability, applications, and limitations of the test method.

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TABLE 1.1. LABORATORY CHARACTERIZATION TESTS FOR ANTIMISTING FUEL

REPORT SECTION	AMK CHARACTERIZATION TESTS	UNDEGRADED AMK	DEGRADED AMK	APPLICATION COMPOSITION AMK	COMPOSITION FM-9 SLURRY
1	Filter Ratio	X	o		
2	Cup	X	o		
3	Turbidity	X			
4	Degradability	o			
5	Transition Velocity		o		
6	Pump Filtration		o		
7	Gel Permeation Chromatography		o		
8	Die Swell	o			
9	Low Temp Gel Formation	o			
10	Inertial Rheometric Pump	o			
11	Viscosity and Viscosity Ratio	o			
12	Solids				o
13	Water Content Determination			o	
14	Glycol Measurement			o	
15	Amine Measurement			o	
16	Density Determination			o	
17	Particle Size Evaluation				o
18	Viscosity Determination				o

X – PRIMARY QUALITY CONTROL TEST

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With the development of FM-9 slurry for the inline blending process and the production of various types of FM-9 slurries, it became important to standardize FM-9 slurry. The FAA, in conjunction with ICI, developed a set of specifications for FM-9 slurry (Avgard ®), using several of the laboratory characterization tests as specified in this report. ICI used these specifications to produce an FM-9 slurry with repeatable handling properties and repeatable antimisting performance in the resulting antimisting fuel. FM-9 slurry specifications (appendix A) include the solids test and the viscosity determination test.

When the antimisting fuel program progressed into the full-scale validation phase with flight testing and the CID, a set of specifications were necessary to evaluate the antimisting fuel that was inline blended into the test aircraft to determine if it had acceptable antimisting properties. A set of specifications were also necessary to evaluate the level of AMK degradation produced by the flight test degrader to determine if the degraded AMK was adequate for engine operation. Based on previous results from laboratory characterization tests specified in this report, a set of specifications was developed for determining acceptable quality AMK and adequate degrader performance. The specifications for acceptable quality AMK (appendix B) include the following laboratory characterization tests: (1) filter ratio test, (2) cup test, (3) turbidity, (4) solids test, and (5) water content. Degrader performance specifications (appendix C) include the filter ratio test and the cup test.

2. LABORATORY CHARACTERIZATION TESTS.

2.1 FILTER RATIO TEST.

2.1.1 Background and Theory.

Developed by the RAE, the filter ratio test was designed as a standardized filtration test to measure intentional degradation of AMK. The filter ratio apparatus is primarily a viscometer that compares the viscosity of Jet A to the viscosity of AMK, yielding a dimensionless number called the filter ratio.

During the early stages of the AMK program the filter ratio test was used to evaluate the performance of various types of degraders by measuring the amount of degradation produced. It was later determined that the filter ratio test was also a useful quality control test for undegraded AMK. The filter ratio test showed repeatable results for undegraded AMK produced with the same polymer batch. With the advent of the inline blending process, filter ratio test results were used to assess the developing equilibration of inline blended AMK.

Since the filter ratio test became standard for all AMK analysis as the primary method of measuring viscosity properties, the standard test procedure was improved at the 9th U.S./U.K. Technical Group Meeting on AMK.

2.1.2 Experimental.

2.1.2.1 Apparatus.

The filter ratio apparatus is shown in figure 2.1. The filterability of an AMK sample is measured by recording the time a precise quantity of fuel at a constant
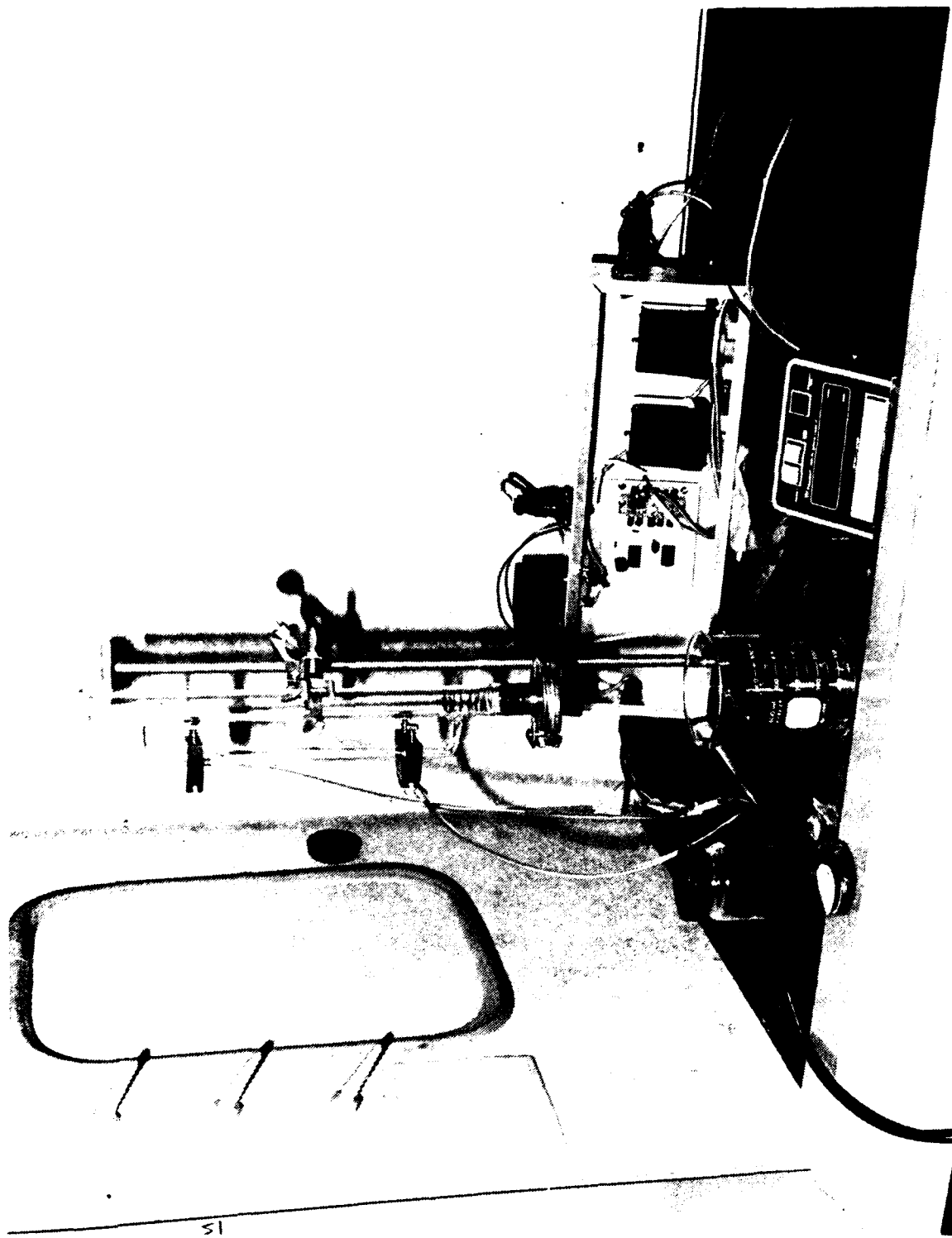
FIGURE 2.1 FILTER RATIO APPARATUS

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fuel temperature under a repeatable gravity head takes to flow through a filter of known dimensions. The AMK flow time compared to the base Jet A flow time, which the AMK was blended from, is known as the filter ratio.

$$\frac{\text{flow time of AMK (sec)}}{\text{flow time of base Jet A (sec)}} = \text{Filter Ratio (FR)}$$

A large filter ratio versus a small filter ratio indicates an AMK with a higher viscosity.

During the latter stage of the AMK program an electronic timer with optical sensors located at the timing reference points shown in figure 2.2 was used to measure the flow times. This improved the accuracy and repeatability of the filter ratio test.

2.1.2.2 Equipment Specifications.

1. Filter Ratio Apparatus - manufactured to the specifications shown in figure 2.2.

2. Viton O-rings - 28 mm diameter, 3.0 mm thick

3. Rapid Release Clamp - Nos. KF 20/25, made by Leybold-Heraeus, Monroeville, PA

4. Electronic Timer - assembled from the following items:

   a. sight glass scanners - No. S19101 (2 required, see note)

   b. photo electric control - No. R40100 (2 required, see note)

   c. timer - start/stop, 110 volt, capable of reading tenths of seconds

NOTE: Item manufactured by Skan-A-Matic Corporation, Elbridge, NY.

5. Cork or Rubber Stopper

6. Filter Disks - 1.5-inch diameter with the following specifications made by Tetko, Inc., Elmsford, NY:

   a. pore size - 16 to 18 micron (nominal)

   b. material - twilled dutch weave stainless streel cloth

   c. mesh - 165 X 1400

   d. wire diameter - .0028" warp diameter X .0016" weft diameter

2.1.2.3 Procedure.

1. Bring Jet A fuel and AMK samples to 20°C +2°C by placing sample containers in a water bath.

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25 mm | 12 mm

85 mm

1st TIMING REFERENCE POINT

170 mm

2nd TIMING REFERENCE POINT

115 mm

GLASS METAL JOINT

CLAMP

GLASS METAL JOINT

SOLDER FILLET

O- RING GROOVE 2mm DEEP

6.5 mm

VITON-O-RING
1.0 mm SQUARE

37 mm DIAM.
FILTER

80-85°

6.5 mm

40 mm

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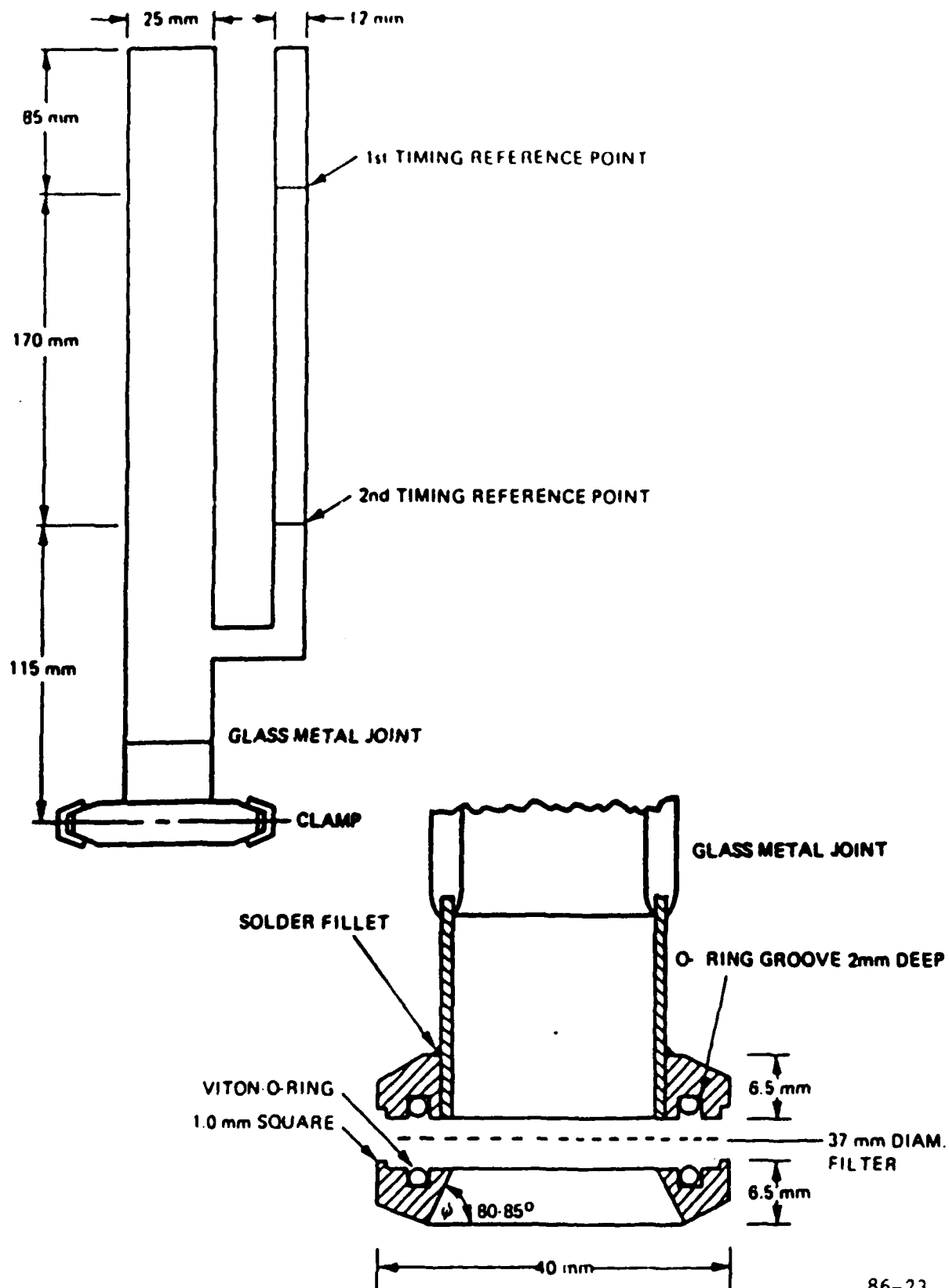
FIGURE 2.2   DESCRIPTION OF FILTER RATIO APPARATUS

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2. O-rings should be properly seated in both the upper and lower filter plates.

3. Place a new filter disk in the lower filter plate and align it with tne upper filter plate. Position both plates together and attach quick release clamp.

4. Insert a stopper in the bottom of the orifice of the lower filter plate.

NOTE: The stopper must not contact the filter disk.

5. Tilt the apparatus slightly and pour the Jet A slowly down on the side of the tube. As the tube is filling, return it to a vertical position and fill until Jet A just begins to spill into the overflow. NOTE: The Jet A used should be the same as used to blend the AMK.

6. Allow Jet A to stand for 60 seconds or until all visible air bubbles have risen to the top before proceeding. NOTE: If stopper should come out prematurely, the procedure must be repeated with a new filter disk.

7. Reset timer to zero. NOTE: Timer is activated while filling as well as upon empting of device.

8. Place beaker under apparatus. Remove the stopper with a twisting motion so as not to disturb the fuel sample. The electronic timer times the passage of fuel meniscus between the two timing marks. Record time.

9. Allow all the fuel to drain.

10. Using the same filter disk, repeat steps 4 through 9 with the AMK sample.

11. Disassemble the filter holder and inspect filter disk. Note the presence of any unusual upstream or downstream gel.

12. Discard filter disk, and clean apparatus with solvent.

13. Calculate the filter ratio.

$$FR = \frac{TIME\ AMK\quad (SEC)}{TIME\ JET\ A\quad (SEC)}$$

2.1.2.4 Results and Discussion.

The filter ratio test is one of the primary quality control tests for AMK and has become standard for all AMK analyses. The filter ratio apparatus primarily compares the viscosity of AMK to the viscosity of Jet A and, therefore, acts as a viscometer. Filter ratio measurements provide information on the level of fire protection, equilibration characteristics, and the level of degradation of an AMK sample.

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There was some initial concern that imperfect dimensions of the filter disk could change the relative flow resistance of the filters and produce unreliable results from the filter ratio tests. The flow times using different filters and the same Jet A were found to vary from 4.0 to 6.0 seconds. This problem was eliminated by measuring the Jet A flow time each time the test was done and using the Jet A that the AMK sample was blended from. Therefore, the test fuels were exposed to the same flow resistance through the filter, giving corresponding differences in Jet A and AMK flow times. The filter ratio values were unaffected. This also holds true for slight variations in effective filter area.

A batch blended or an equilibrated inline blended 0.3 percent FM-9 AMK normally yields a filter ratio between 35 and 90. Values outside this range could be a result of any one or more of the following: unequilibrated AMK, incorrect polymer concentration, sample degradation, or contamination. The filter ratio test can distinguish inadequate AMK blends that would not have shown up in the cup or turbidity test results (sections 3.2 and 3.3). For example, a developing inline blended AMK sample may have a normal cup and turbidity value but could exhibit an extreme plugging condition in the filter ratio apparatus (FR greater than 100).

Degrader performance is also determined by the filter ratio test. Filter ratio values of 1.5 and below are indicative of highly degraded AMK. A maximum filter ratio value of 1.2 has been established as an acceptable level of degrader performance. However, the filter ratio test is insensitive at values less than 1.2. Tests have shown that the flow time (4 to 6 seconds) for highly degraded AMK samples is too short. Other tests, such as the pump filtration test or transition velocity test (sections 3.5 and 3.6), must be employed to effectively evaluate very highly degraded AMK.

2.2 ORIFICE FLOW TEST (CUP TEST).

2.2.1 Background and Theory.

The orifice flow cup was originally developed for use in the paint process; the cup was later modified by ICI solely for use with FM-9 antimisting fuel. The cup test is one of the primary quality control tests for antimisting fuel. Flow through the orifice flow cup would inherently seem to involve elongational deformations of the fluid. However, since the dimensions of the orifice yield a length to radius ratio (L/R) 3.85, one could expect that shear deformations could occur. Tne effects of shear viscosity on the flow rate through the orifice flow cup is shown by experiments done with Newtonian fluids. A Jet A/mineral oil blend with a shear viscosity of 13.6 centipoise (cP) produced an orifice flow cup measurement similar to that of a 0.3 percent FM-9 AMK out would exhibit no fire protection. Mannheimer of SWRI also points out that the shear rate developed in the orifice flow cup is well above the critical shear rate established for shear thickening behavior of AMK (reference 2).

2.2.2 Experimental.

2.2.1 Apparatus.

The orifice flow cup is shown in figure 2.3 and is similar to a type A cup that is specified in tne British Standard Specification for Flow Cups and Methods of Use, except that the orifice diameter is changed to increase the cup's

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FIGURE 2.3   ORIFICE FLOW CUP TEST APPARATUS
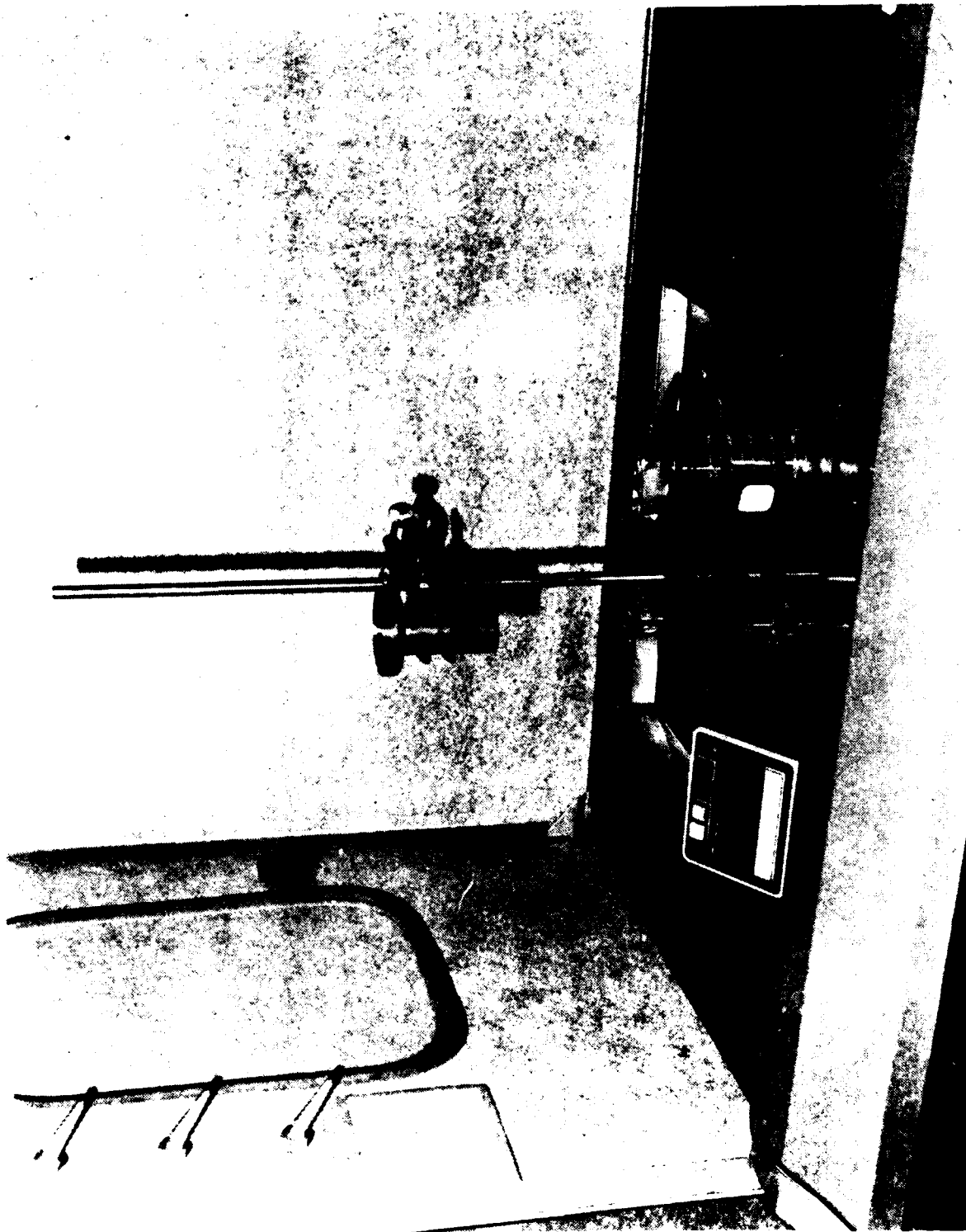
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sensitivity for use with FM-9 AMK. The cross section of the orifice flow cup is shown in figure 2.4. AMK at a specific temperature passes through the orifice of the flow cup under a known, but diminishing, pressure head and is collected for a specified time. The collected volume of AMK is then measured in milliliters.

2.2.2.2 Equipment Specifications.

1. Orifice flow cup, similar to that of type A cup, as specified in British Standard Specification for Flow Cups and Methods of Use, Method Identification B.S. 1733, except that the orifice diameter shall be 0.026 inches +0.0005 inches. See the specification for the flow cup shown in figure 2.4.

2. Timer (seconds), capable of reading tenths of seconds

3. Graduated cylinder, 10 ml graduated in 0.2 ml divisions

4. Magnifying glass or eye piece

2.2.2.3 Procedure.

1. Clean the flow cup and graduated cylinder immediately before each test.

    a. Wash cup with solvent (1,1,1 - Trichloroethane).

    b. Inspect orifice with magnifying glass.

    c. Clear any obstruction in orifice with nylon or coarse animal hair bristle (corn broom bristle also works). Do not use any hard or mallic probe to clean the orfiice.

    d. Rinse again with solvent.

    e. Air dry.

    f. Hold cup to a light source and inspect with glass.

2. Check flow cup performance at the beginning and end of each test series.
    a. Clean as described in item 1.

3. Adjust temperature of sample to 20°C +2°C by placing in water bath. Avoid rapid heating or cooling of sample.

    a. Measure flow rate of Jet A by using the procedure described below. Flow rate for Jet A should be 7.6 to 7.8 ml/30 seconds.

4. Place the flow cup on the stand in a place free from drafts. Make sure that the cup is at sufficient height above the bench to permit the introduction of the graduated cylinder below it.

5. With the orifice closed by one's finger, fill the cup with a bubble free sample, until it just begins to spill into the overflow. Pour slowly down the sides of the cup to minimize formation of air bubbles or gellation.

FIGURE 2. [illegible caption]
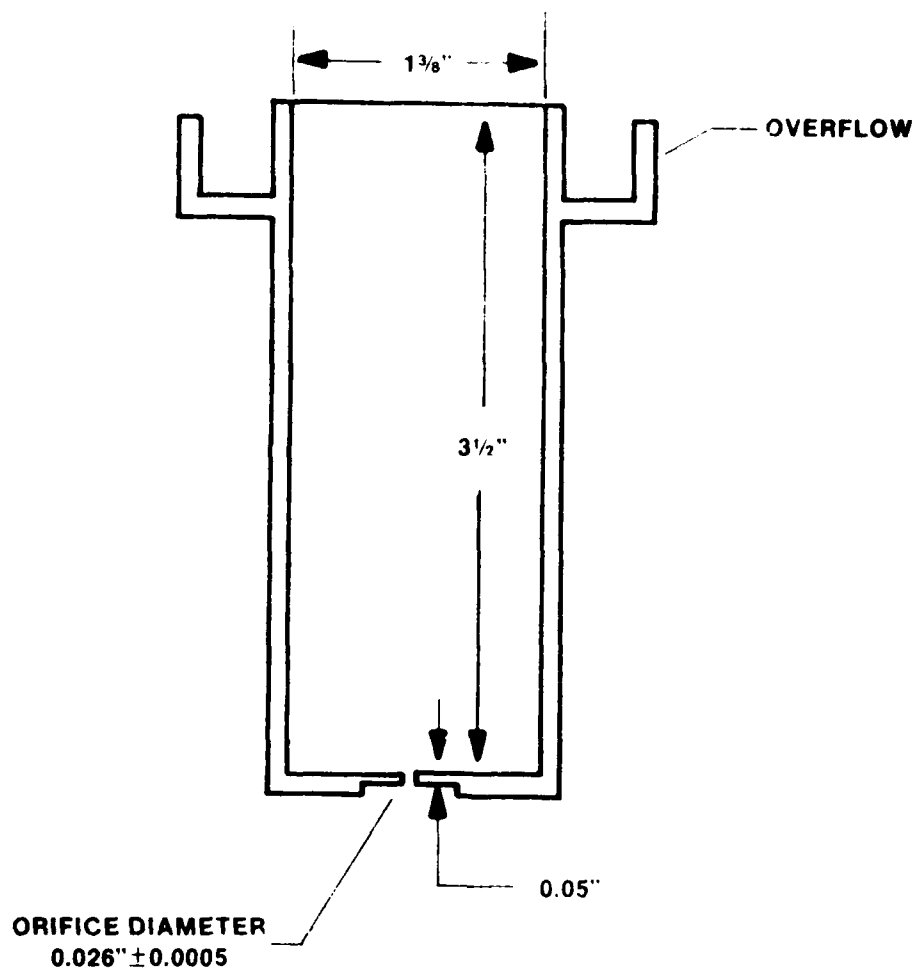
6. Level orifice cup, zero timer, place beaker under cup.

7. Start timer and hold finger over orifice for 30 seconds.

8. At exactly 30 seconds, remove finger and allow fuel to flow into beaker for 30 seconds.

9. At exactly 60 seconds (total elapsed time), introduce the dry graduated cylinder beneath the cup.

10. Collect material from the orifice for exactly 30 seconds (90 seconds total elapsed time).

11. Record the amount of sample collected in the graduated cylinder to the nearest 0.1 ml.

12. Repeat the procedure until consecutive tests give three values within 0.1 mls of each other. Report the average of the three values.

Care of Cup:

1. Clean cup and graduated cylinder immediately after each test as described in item 1 above.

2. Do not wipe inside of cup or around orifice with fibrous material.

3. When not in use, store cup with orifice immersed in Jet A.

2.2.2.4 Results and Discussion.

The orifice flow test is one of the primary quality control tests for inline blended AMK and batch-blended AMK as well as being a good indicator of the degradation level of a degraded AMK sample. The orifice flow cup measures shear viscosity which makes the cup primarily a viscometer (reference 2). In use with 0.3 percent FM-9 AMK it measures the shear viscosity above the critical shear rate.

Generally, an undegraded equilibrated inline blended 0.3 percent FM-9 AMK exhibits an orifice flow cup value between 1.7 and 2.3 ml/30 sec depending on the polymer lot. Values outside of this range are indicative of: unequilibrated AMK, unclean orifice flow cup, incorrect polyer concentrations, AMK degradation during sampling or sample contamination. Degraded AMK yields an orifice flow cup value between 2.5 and 7.0 ml/sec, depending on the level of degradation and highly degraded AMK produces values greater than 7.0 ml/30 sec. A minimum orifice flow cup value of 7.0 ml/30 sec has been established as an acceptable level for degrader performance.

The developing dissolution of inline blended AMK can be tracked using orifice flow cup results, and orifice flow cup values can quickly indicate poor quality AMK.

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2.3 TURBIDITY (CLARITY) TEST.

2.3.1 Background and Theory.

Turbidity is a term given to an optical property of a liquid and is defined as an appearance parameter. Turbidity is caused by the presence of suspended matter in in host liquid. In the case of AMK, turbidity can be caused and affected by such things as partially swollen polymer, gels, wet and dry particles, high polymer concentration, turbidity of the base Jet A, and the water content of the base Jet A or AMK (reference 3). The laboratory device used to measure turbidity is a Nephelometer.

2.3.2 Experimental.

2.3.2.1 Apparatus.

A Nephelometer, such as the one shown in figure 2.5, produces an intense beam of light which is passed through a standard 30 ml sample bottle that contains the liquid to be measured. Suspended material in the liquid deflects a small portion of the light out of the main light beam. The light that is deflected at right angles is measured by a sensitive light detector, and the resulting electrical signal is amplified to provide output which is displayed on a digital voltmeter in nephelometric turbidity units (figure 2.6). This reading is calibrated to the concentration of suspended matter.

2.3.2.2 Equipment Specifications.

Any apparatus designed for the purpose of measuring the turbidity of a liquid can be used to measure the turbidity (clarity) of an antimisting fuel or Jet A sample. During the course of the AMK program, the FAA, ICI, and its contractors used several different models of Nephelometers. All of the Nephelometers used produce turbidity measurements that are reliable and relatable if the Nephelometers are accurately calibrated before use. The Nephelometers used by the FAA, ICI, and JPL are specified in Section 2.3.2.2.1 through 2.3.2.2.3.

2.3.2.2.1 Nephelometer (Used by the FAA).

1. Laboratory Nephelometer, Model 21, manufactured by Monitek, Inc., Hayward, California, with the following specifications:

Ranges:	0-1.99, 0-12.9, and 0-199 NTU switch selectable (Decimal point is automatically set on the digit display.)
Accuracy:	+1 digit of calibrated span. (Overall accuracy dependent on calibration standard.)
Reproducibility:	+1 digit
Sensitivity:	+0.005 NTU
Stray light:	+0.01 NTU (Lowest range with sample cover installed)
Response Time:	100 milliseconds (63% change)

FIGURE 2.5 NEPHELOMETER

Sample Required: 25 milliliters minimum

Sample Bottle: 25 mm x 95 mm vial (30 ml capacity)

Display: 2 1/2 digit, digital readout

Recorder Output: 0-1 milliampere DC

Type of Lamp: Incandescent (Halogen Cycle Type)

Warm-up Time: 5 minutes

Dimensions: 11" x 11" x 3" (28 cm x 28 cm x 7.6 cm)

Power Requirements: 110V, 50/60 Hz (25 Volt-Amps)

    2.   Turbidity standard 10 NTU, catalog number 2480, manufactured by the Hach Company, Loveland, Colorado 80539.
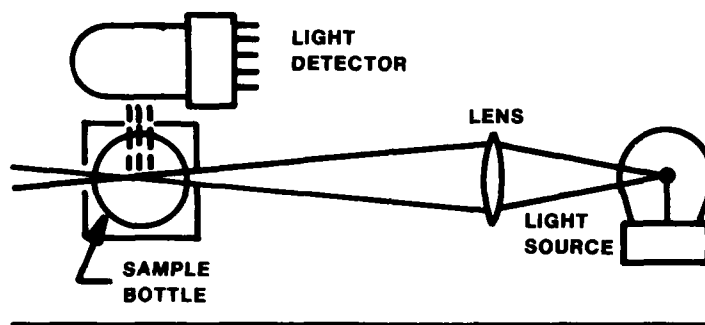
FIGURE 2.6   SCHEMATIC OF NEPHELOMETER OPERATION

2.3.2.2.2 Nephelometer (Used by JPL).

Turbidmeter, model DRT100, manufactured by H. F. Instruments

2.3.2.2.3 Nephelometer (Used by ICI).

Turner Designs Nephelometer, Model 40-002, manufactured by Turner Designs, Mountain View, California.

2.3.2.3 Procedure (Monitek, Model 21, Nephelometer).

    1.   Turn on the power and lamp switches and allow the Nephelometer to warm up for 15 minutes.

    2.   With sample chamber empty and cover in place, set the range switch at 20 NTU and turn the zero adjust knob for a 0.0 reading on the digital voltmeter display.

    3.   Install the 10 NTU turbidity standard into the sample chamber and replace cover.

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4.   Adjust the standardize control knob for a digital display reading equal to the value of the turbidity standard.

5.   Remove the turbidity standard from the sample chamber and insert a sample holder bottle filled 80 percent or more with the fluid to be measured and replace cover.

6.   Record the value displayed on the digital voltmeter to the tenth of an NTU, approximately 15 to 20 seconds after insertion.   (Turbidity will change as sample is warmed in Nephelometer).

7.   Remove sample, drain and clean vial with solvent.

8.   According to the manufacturer, it is not necessary to check the zero and standardize the Nephelometer before each reading, and in fact, if the instrument is functioning properly, frequent calibration checks are not necessary.   However, the validity of the test data depends on the calibration and until the operator has gained sufficient, implicit confidence in this instrument's stability, frequent calibration checks are recommended (preferably before each sample reading).

9.   Operational Notes

a.   Two horizontal bars on the digital display indicate an over-range condition, switch to the 200 range.

b.   Turbidity standards should be stored in an opaque container.

c.   Sample holder bottles and calibration standards should be free of finger prints.

2.3.2.4   Results and Discussion.

NTU values between 4 and 20 are normal for 0.3 percent FM-9 AMK, although higher readings have occurred when there has been a problem in the blending or storing of the AMK.   A high NTU value is indicative of polymer which has not yet fully dissolved, and a low NTU value is indicative of an equilibrated AMK sample. Although there is no absolute NTU value, an equilibrated AMK sample normally will yield an NTU value between 5 and 10.   Also, the developing equilibration of an inline blended AMK sample from the same polymer lot should track the same (i.e., turbidity versus time) and reach the same turbidity equilibrium value.   The turbidity of developing AMK always exhibits a decreasing NTU behavior.

The development or dissolution of freshly blended AMK can be followed by using Nephelometry.   The turbidities of developing AMK samples exhbit asymptotic behavior.   A typical plot of turbidity versus time is shown in figure 2.7.   The rate of change of turbidity data provides an indication of the state of equilibrium in which an AMK sample is in.   However, the turbidity equilibrium value is reached several hours before the degradability equilibrium (maximum degradability) is reached.

17
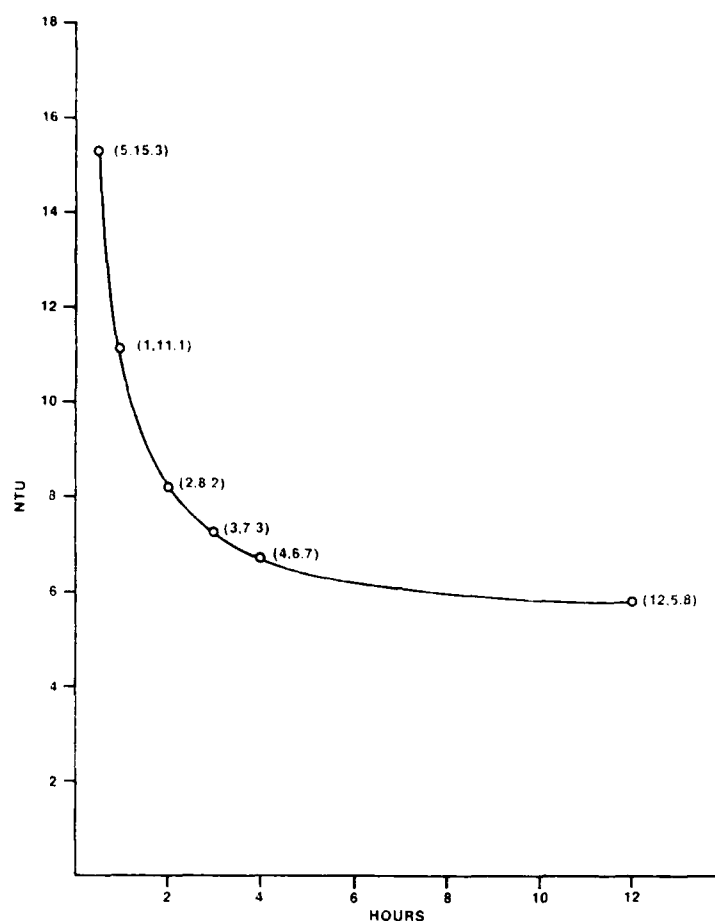
FIGURE 2.7   TURBIDITY OF CID BLEND

There are several limitations of turbidity measurements. For example, turbidity data does not indicate developing AMK blends which exhibit filter plugging tendencies. Also, there seems to be no relationship between the degradation level of an AMK sample below a filter ratio of 5 and turbidity measurements.

2.4   DEGRADABILITY TEST.

2.4.1   Background and Theory.

This test was developed by JPL as a means to determine the degradability of developing inline blended AMK on a laboratory scale. It is important to determine the development time at which an inline blended AMK fuel can be degraded to an acceptable level for aircraft operations (filterability, combustibility). The degradability test provides an assessment of the degradability and the degradation development of an AMK fuel on a laboratory scale.

2.4.2 Experimental.

2.4.2.1 Apparatus.

The apparatus is basically a soda fountain type mixer as pictured in figure 2.8. The mixer produces a relatively repeatable mixing revolutions per minute (RPM) using 110-volt Alternating Current (A.C.) power that is readily available. Also, the mixing time and volume are held constant, therefore, each AMK sample is degraded at relatively the same specific power.

2.4.2.2 Equipment Specifications.

1. Hamilton Beach Scovill Mixer, Model No. 936-2

2. Stainless steel mixing cup for use with above mixer.

2.4.2.3 Procedure.

1. Pour 300ml +5 ml of undegraded AMK into the mixer's stainless steel container.

2. Set mixer speed setting to LOW.

3. Place stainless steel container with AMK into mixer.

4. Run mixer at LOW speed for 5 minutes.

5. After mixing for 5 minutes, determine filter ratio as per Section 2 of this report.

NOTE: The filter ratio measurement should be made immediately after the 5-minute mixing operation to avoid the misleading filter ratios associated witn freshly blended and degraded AMK.

2.4.2.4 Results and Discussion.

Although the degradability test has been used on a limited basis, AMK samples can be directly compared with other AMK samples or developing AMK samples can be tracked to determine the time when maximum degradability is reached. Measured AMK samples are degraded in the mixer for a specific time with relatively the sample specific power input. Generally, an AMK sample which yields a filter ratio of 3 or less after the degradation test is considered to be adequately equilibrated.

2.5 TRANSITION VELOCITY TEST.

2.5.1 Background and Theory.

The transition velocity (V_T) test method was developed by PWA as a more sensitive method (than filter ratio, cup, etc.) for evaluating degradation levels of antimisting fuels. Also, the V_T test method provided a relatively fast means of producing repeatable results of degradation levels.

86-013

86-23

FIGURE 2.8 DEGRADABILITY TEST APPARATUS

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PWA found that when AMK flow through a filter is plotted as a function of increasing differential pressure (Δ P) across the filter, there is an increase in flow resistance which occurs at a specific flow rate or velocity, known as the transition velocity (reference 4). The transition velocity is directly related to the degradation level of the AMK. The higher the transition velocity, the closer the AMK is, in properties, to Jet A fuel. These points are illustrated in figure 2.9.
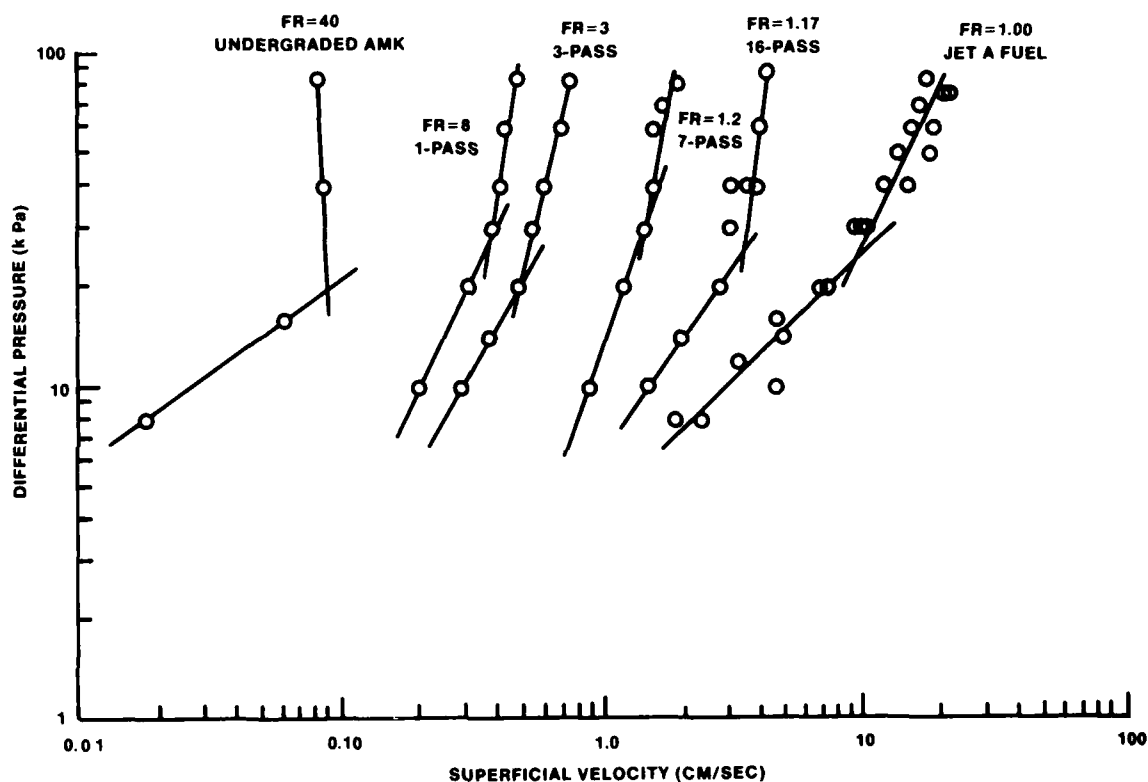
FIGURE 2.9   TRANSITION VELOCITY AND FILTER RATIO COMPARISON

2.5.2   Experimental.

2.5.2.1   Apparatus.

The apparatus is shown in figure 2.10.   Basically, the V_T test apparatus is a millipore filtration apparatus with a ball valve added between the vacuum flask and the filter support (reference 5).   The millipore filtration apparatus is a three-piece glass filter funnel.   The fine filter in the millipore apparatus is replaced by a 16-mesh wire screen in the V_T test apparatus.   The transition velocity test measures filterability by drawing a measured volume of fuel through a filter of known dimensions under different vacuum levels and recording the time

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for this measured volume to pass through the filter. Using the data, a fuel velocity is calculated and plotted against the vacuum level. The plot result is two intersecting, straight lines, the point of intersection is defined as the transition velocity.

FIGURE 2.10   PWA TRANSITION VELOCITY APPARATUS

The V_T unit, as designed, contained serveral inadequacies in the apparatus. AMK samples leaked out of the glass-to-glass joint on the millipore filtration apparatus, and there were no clear points to start and stop the timing of the sample due to the large meniscus of the sample and the velocity of the sample when it passed through the filter at the stop point. Since the vacuum gauge supplied with the pump had a small scale and the readings fluctuated, it was difficult to obtain accurate and repeatable readings.

The FAA Technical Center modified the V_T apparatus (figures 2.11 and 2.12) to incorporate a modified filter ratio apparatus with electronic timers in place of the millipore filtration apparatus and a 6-inch diameter vacuum gauge with inline damping to smooth out vacuum fluctuations.

During the initial development of the V_T test, the apparatus was tested using 8 μm Nuclepore paper filters, 40 μm engine fuel pump paper filters, 10 μm woven metal filters, and 17 μm woven metal screens. The Nuclepore paper filter was chosen for use in the V_T apparatus because of the well defined and consistent characteristics relative to pore size, geometry, and pore density (reference 4). The Nuclepore filter is a polycarbonate filter with a pore diameter of 8 μm pore density of 1×10^5 pores/cm^2 and a nominal filter thickness of 10 μm.

22
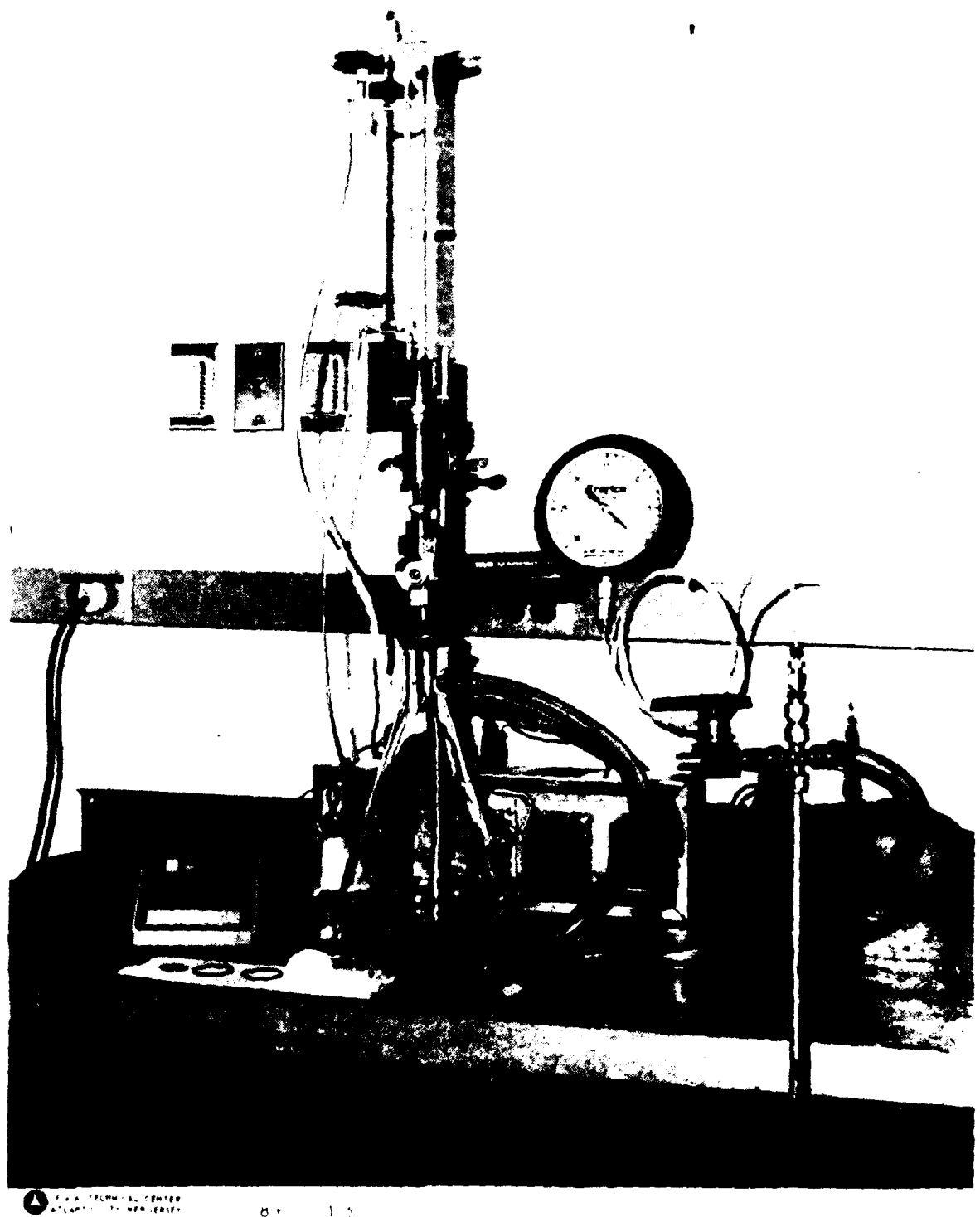
FIGURE 2.11 FAA TRANSITION VELOCITY APPARATUS
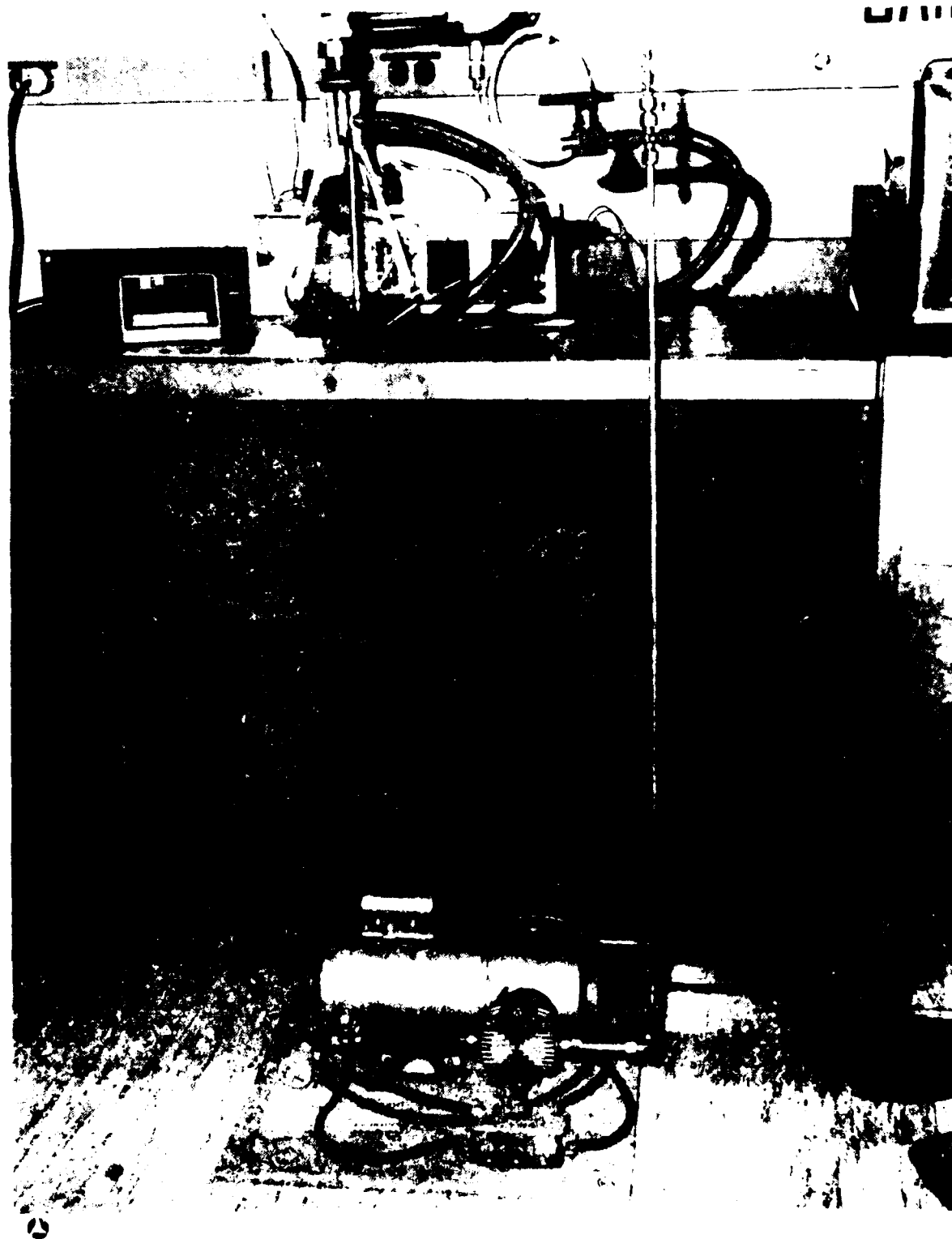
2.5.2.2 Equipment Sp.cifications.

2.5.2.2.1 PWA V_T Apparatus.

    1. 200 ml pyrex filter flask, No. 5340.

    2. Millipore filtration apparatus, No. 4. modified with larger sample reservoir (4 inches higher).

    3. 47 mm diameter, 8 μm pore size, Nuclepore polycarbonate membrane filters.

    4. 16-mesh wire screen filter support, see figure 2.10.

    5. 1/2-inch ball valve.

    6. Vacuum pump, capable of producing 0 to 20 inches of Hg. Gast vacuum pump, model 0211-V45F-68CX.

    7. Timer, capable of reading tenths of seconds.

    8. Miscellaneous, pipe fittings, rubber stopper and tygon tubing 3/8" I.D., 1/4" wall thickness.

2.5.2.2.2 Modified V_T Apparatus.

    1. 2000-ml pyrex filter flask, No. 5340.

    2. Modified filter ratio apparatus, see figure 2.11.

    3. Electronic Timer setup for filter ratio apparatus, see filter ratio apparatus equipment specifications.

    4. 1/2-inch ball valve.

    5. Vacuum pump, capable of producing 0 to 20 inches of Hg, gast vacuum pump, model 4VCF-10-M400X.

    6. 16-mesh, 1 1/2-inch diameter filter support.

    7. 47 mm diameter, 8 μm pore size, Nuclepore polycarbonate membrane filter.

    8. 3/8-inch swage lock ball valve.

    9. Miscellaneous, pipe fittings, 3/8-inch tubing and fittings, rubber stopper and tygon tubing 3/8" I.D., 1/4" wall thickness.

2.5.2.3 Procedure.

2.5.2.3.1 PWA V_T Procedure.

1. Assemble the apparatus shown in figure 2.10. The device is basically a millipore filtration apparatus with a ball valve added between the vacuum flask and the filter support. In addition, the fine wire mesh filter support is replaced with a 16-mesh wire screen.

2. Place a 47 mm diameter, 8 μm pore size Nuclepore filter on the support and close the valve to the flask.

3. Add 300 ml of the test fuel to the reservoir. Turn the vacuum pump on and adjust the vacuum level to 2 inches of mercury with the valve closed.

4. Simultaneously, open the valve and start the timer. Rapidly make any fine adjustment necessary to keep the vacuum at the desired level. Record the time to the nearest tenth of a second for all the fuel to pass through the filter.

5. Using the same filter, repeat steps 3 and 4 at vacuum levels of 3, 4, 10, 15, 17.5, and 20 in Hg vacuum.

6. Prepare a plot of flow rate in cc/sec-cm^2 versus vacuum level. Plot a straight line through the first three points and another straight line through the top three points. The flow rate where the two lines intersect should be reported as the transition velocity in cc/sec-cm^2.

7. Replace the filter for each additional fuel tested.

2.5.2.3.3 Modified V_T Procedure.

1. Assemble the apparatus shown in figures 2.11 and 2.12.

2. Place a Nuclepore polycarbonate membrane filter on the 16-mesh wire screen filter support and assemble the filter ratio tube. Close the ball valve between the sample reservoir and the flask.

3. Set the sight glass scanners so that the sample size between the two timing marks is 135 ml which will be used in the calculation of the superficial velocity. Fill the filter ratio tube with the test fuel until it overflows into the catch basin.

4. Turn on the vacuum pump and adjust the vacuum level to 2 inches of mercury with the ball valve closed.

5. Open the ball valve after the fuel sample has stabilized for one minute. Let all the fuel pass through the filter and record the time from the electronic timer to the nearest tenth of a second.

6. Close sample valve immediately after all fuel passes through filter.

7. Using the same filter, repeat steps 3, 4, and 5 at vacuum levels of 3, 4, 10, 15, 17.5, and 20 in. Hg vacuum.

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8. Prepare a plot of flow rate in $cc/sec-cm^2$ versus vacuum level. Filter area for use in the computation is 4.45 cm^2. Fuel volume for use in this computation is 135 cc.

$$\text{Superficial Velocity} = \frac{\text{fuel volume}}{\text{time x filter area}}$$

Plot a best fit straight line through the first three points (2, 3, and 4 inches Hg) and another through the top 3 points (15, 17.5, and 20 inches Hg). The flow rate where the two lines intersect is reported as the transition velocity in $cc/sec-cm^2$. See sample plot in figure 2.13.

9. Replace the filter for each additional fuel sample tested.

2.5.2.4 Results and Discussion.

The test procedures call for the PWA V_T apparatus to have a 300 ml sample size and the modified V_T apparatus to have a 135 ml sample size. The flow per cm^2 of filter area can be determined as shown below:

PWA V_T Apparatus   Modified V_T Apparatus

$$\frac{300 \text{ ml}}{8.5 \text{ cm}^2} = 35.0 \text{ ml/cm}^2 \qquad\qquad \frac{135 \text{ ml}}{4.45 \text{ cm}^2} = 30.3 \text{ ml/cm}^2$$

Thus, even though the modified sample size is 65 percent less than the PWA sample size, there is only a 15 percent difference in the flow per cm^2 of filter area due to the smaller effective filter area of the modified V_T apparatus.

As shown in figure 2.9 and according to PWA V_T literature, Jet A fuel should produce a transition velocity of 9 $\pm$2 $cc/sec-cm^2$ (reference 6). However, no transition velocity for Jet A using either the modified or the PWA VT apparatus, could be obtained by FAA personnel. FAA personnel found that when Jet A test results were plotted (flow rate versus vacuum level), a linear plot was obtained over a range of differential pressures from 2 to 20 inches of Hg with superficial velocities of 4.8 to 26.3 cm/sec.

This test is not suitable for use with AMK samples with filter ratios greater than 1.5. Using this apparatus on AMK samples with filter ratios greater than 1.5 may result in filter plugging and, therefore, inaccurate test results.

The transition velocity test method has only been used on a limited basis but has been shown to have the ability to detect changes in AMK fuels at high levels of degradation where the filter ratio test method loses accuracy.

A transition velocity of 2 to 3 cm/sec was established by PWA as an acceptable level of degrader performance for the CID (reference 7).

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SAMPLE NO. N17CL
F/R 1.3
CUP 5.8
VT 2.5

PT. NO.	X	Y
1	1.0100	2.0000
2	1.3000	3.0000
3	1.5000	4.0000

COEFFICIENTS

B(0) = -1.549240869E+00
B(1) = 3.779069800E+01

R SQUARE = 0.999897415

PT. NO.		
1	2.6800	15.0000
2	2.7600	17.5000
3	2.9100	20.0000

COEFFICIENTS

B(0) = -8.642441949E+01
B(1) = 3.779069800E+01

R SQUARE = 0.382558146

SUPERFICIAL VELOCITY, cc/sec-cm2 (cm/sec)

IN Hg
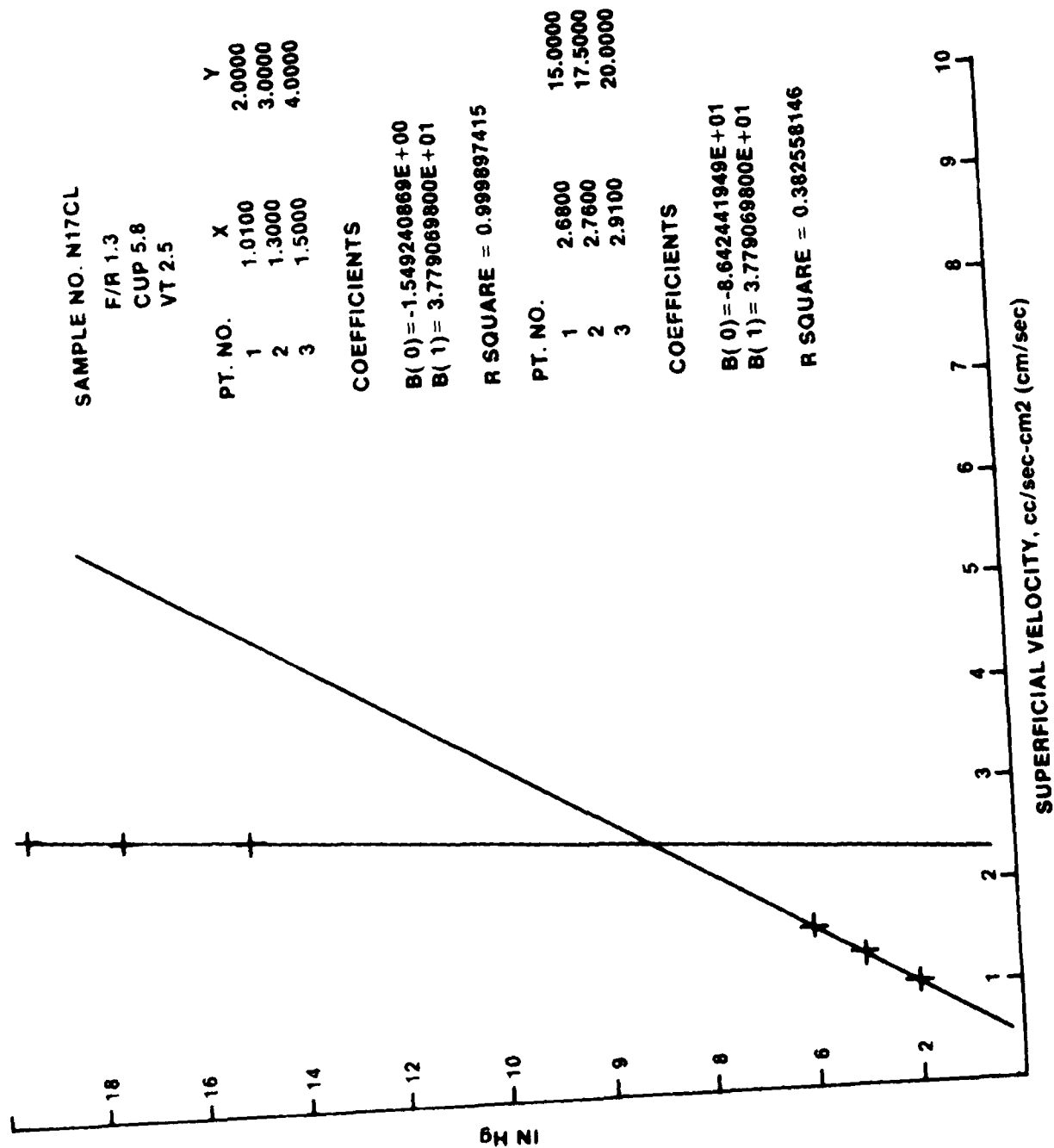
FIGURE 2.13 SAMPLE TRANSITION VELOCITY PLOT

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2.6 PUMP FILTRATION TEST.

2.6.1 Background and Theory.

The pump filtration test was developed by SWRI in order to characterize the
filterability of highly degraded AMK with a filter ratio less than 1.2, due to
the insensitivity of the filter ratio test at low values. A degraded AMK sample
characterized by the standard filter ratio test produced a FR of 1.4 using the
standard 16-18 micron stainless steel filter disk with an effective filter area
of 5.0 cm^2. Experiments have shown that by decreasing the effective filter area
of the FR apparatus, the same degraded AMK sample showed a steady increase in the
resulting filter ratio (reference 8). These results are shown in figure 2.14.
The results show that the flow time of 4 to 6 seconds for highly degraded AMK in
the standard filter ratio test is too short to detect gel formation that can
occur within the filter. It would be possible to make a better determination of
the filter ratio by decreasing the effective filter area. This led to the
development of the pump filtration test which simulates the flow conditions that
would be encountered in an aircraft fuel system. Degraded AMK will filter like a
Newtonian fluid (Jet A) below the critical filtration velocity, but above the
critical filtration velocity, filter plugging will occur. The pump filtration
test is able to measure the flow resistance of highly degraded AMK through a
filter as a function of time.
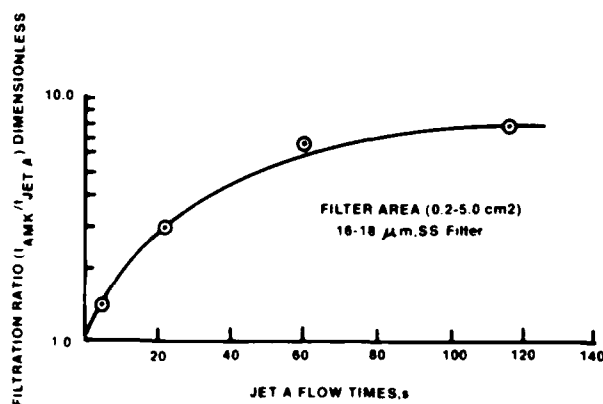
FIGURE 2.14 EFFECT OF FLOW TIME (AREA) ON FILTRATION
RATIO OF DEGRADED AMK

2.6.2 Experimental.

2.6.2.1 Apparatus.

The pump filtration apparatus is shown in figures 2.15 and 2.16. The apparatus
determines the filterability of a degraded AMK sample by measuring the pressure
drop across a filter of known area as a function of time at increasing flow rates
by using a pump to force a fuel sample through the filter. The pressure drop
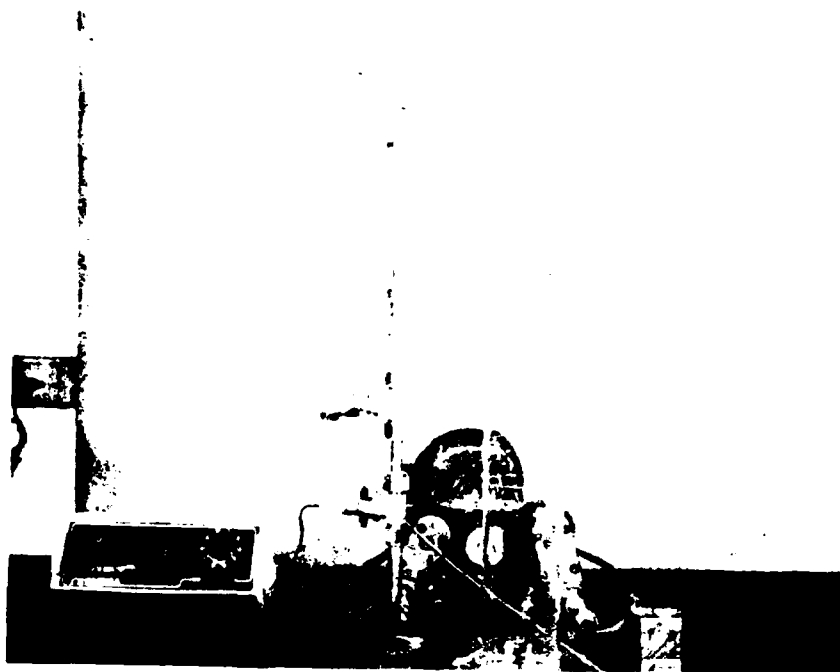
FIGURE 2.15 FULLY ASSEMBLED PUMP FILTRATION APPARATUS
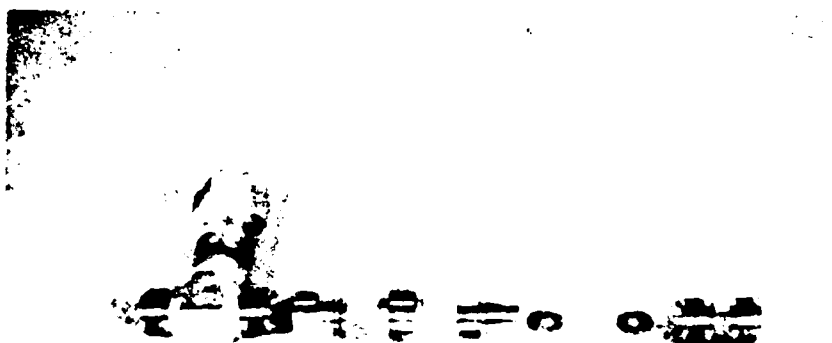
FIGURE 2.16 ASSEMBLY OF PRESSURE TRANSDUCER AND FILTER
AND FILTER BACKUP DISCS

across the filter is measured by a pressure transducer and recorded on a stripchart recorder. A critical filtration velocity (CFV) can then be determined by finding the highest flow rate at which the degraded AMK sample can flow through the filter without resulting in a steady pressure rise in a specified time interval and dividing by the filter area. Below the critical filtration velocity, the pressure with an AMK sample is only slightly greater than with Jet A and remains constant over the specified time interval, and above the CFV, the pressure continues to rise.

2.6.2.2 Equipment Specifications.

1. Metering unit, Zenith, Type 1ZM-4890.

2. Variable speed pumps, Zenith, 0.584 cc/rev, and 2.92 cc/rev.

3. Pressure transducer, CEC, Type 4-313, 0-10 psid.

4. Stripchart recorder, single pen.

5. Amplifier, Action Pak, Model 4051-203.

6. 1/2-inch stainless steel tubing:

   a. 20 cm long with a 90° bend.

   b. 10 cm long with square, polished ends. Note the end of tubing must be polished so as to fit flush against the backup discs.

   c. 5 cm long with square, polished ends.

   d. 5 cm long with one flared and one square, polished end (4 each).

7. 1/2-inch stainless steel union (2 each).

8. Stainless steel female branch tees with 1/2-inch tubing and 3/8-inch pipe connections (2 each).

9. Stainless steel reducing unions, 1/2 inch to 1/4 inch (2 each).

10. Stainless steel tubing 1/4 inch, 3 cm long with square, polished ends.

11. Hose clamps 1/2 inch (3 each).

12. Tygon tubing 1/4 inch, 30 cm long.

13. Backup discs (D = 1.27 cm L = 0.18 m):

   ID = 0.356 cm (2 each), 0.1 cm^2 effective filter area.
   ID = 0.504 cm (2 each), 0.2 cm^2 effective filter area.
   ID = 0.714 cm (2 each), 0.4 cm^2 effective filter area.

14. Iron-constantine type thermocouple shrouded with 18-inch tubing.

15. Digital readout, Omega, Model 199.

A pump filtration test apparatus was assembled by the FAA Technical Center that was based on the design of the apparatus developed SWRI. There were, however, two differences in the FAA apparatus. First, the pump used to force the fuel through the filter was a Masterflex Progressive Cavity Pump Model Number 7520-20 with a Model Number 7017-21 head made by the Cole-Parmer Instrument Company, Chicago, Illinois. The second difference was the filter holder assembly that was constructed to allow rapid changing of the test filter. Two quick change filter assemblies were constructed. One had an effective filter of 1.28 cm^2, and the other had an effective filter area of 0.32 cm^2. The FAA apparatus was used following the same basic procedures as used for the SWRI pump filtration test, as stated in section 2.6.2.3.

2.6.2.3 Procedure.

1. Sample Preparation - Pour two liters of fuel into a large separatory flask. Place a No. 41 Whatman filter paper in a regular funnel and collect the filtered fuel in a clean container.

2. Cleaning Procedure - After the apparatus has been used with AMK, flush the pump thoroughly with Jet A. The removable components such as unions, sections of stainless steel tubing, backup discs, and transducer adapter are cleaned with trichloretnylene and acetone and then are air dried. The pump should be taken apart after every 5-7 experiments and cleaned.

3. Apparatus Preparation - The fully assembled apparatus is shown in figure 2.15. While satisfactory assembly of the various components might be achieved by different methods, the following method has been found to minimize the entrapment of the air in the system. Connect one end of the 1/2-inch tygon tubing to the suction side of the pump and place the other end of the tube in a clean container that holds approximately 2 liters of fuel. Fill this container with filtered fuel (see item 1 for filtering procedure). Raise the baseplate of the pump slightly to help displace air from the lines as they are filled. Connect the 1/2-inch stainless steel tube with the 90° bend to the discharge end of the pump so that the down-stream end of the tube is vertical. Fill the lines at a relatively high flow rate (30 ml/min). Return the baseplate to its original position, and then connect the thermocouples, pressure transducer, and the piece of 1/2-inch stainless steel tubing with polished end as shown in figure 2.16. Note: The polished end of the tube should be positioned downstream so as to fit against the filter backup disc. Restart the pump, and fill the remainder of the line with fuel.

Place the filter element between the two backup discs and insert all three components into a 1/2-inch union. Almost any type of filter material can be used that has sufficient structural integrity and that can be cut into a disc-shaped element. Set the pump at its lowest output (almost zero flow) and continue to fill the system until a convex meniscus is formed at the top of the tube. This procedure helps to prevent air entrapment below the filter.

Caution: Because of the vertical positioning of the tubing, there is a tendency for fuel to flow back through the pump when it is shut off. Since this may draw air into the system, it is advisable to leave the pump running at a low speed for the remainder of the test.

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Next, attach the union that contains the filter and the filter backup discs to the fuel line and tighten the entire assembly. Also connect the pressure transducer lead to the amplifier and recorder. (The pressure is measured immediately upstream of the filter. Use of the transducer in the differential mode was found to make it difficult to remove air from the system.) To further insure that all the air has been removed, place a rubber stopper in the end of the union and allow tne pressure to build up for a few seconds. Quickly release the pressure by removing the stopper and check for the evolution of bubbles.

Caution: Do not exceed the pressure limit of the transducer (10 psi).

Repeat this procedure two or three times or until no bubbles are observed. Finally, connect the remaining pieces of tubing as shown in figure 2.15.

4. Conducting tne Test – Before conducting experiments with AMK, the system should be checked with Jet A. This will insure that the apparatus has been adequately cleaned (item 2) and that the filter has been properly installed (item 3). For example, the pressure with Jet A should not increase over the 2-minute flow time that is used in this test. If the pressure increases with time, either the fuel is contaminated or the apparatus has not been cleaned adequately. In any event, the apparatus should be recleaned, a new filter installed, and the system retested with a fresh sample of filtered Jet A. If the pressure remains constant but appears to be higher or lower than normal (this can only be determined by experience), the filter should be changed and the system again checked with Jet A. If the results with Jet A prove to be satisfactory, experiments can then be conducted with AMK. It is unnecessary to reclean the apparatus after the experiments with Jet A. The lines are merely drained and then flushed with AMK. Furthermore, the same filter should be used, however, the 1/2-inch union that holds the filter should be removed and the system refilled with AMK according to item 3 to insure complete removal of tne air from the system.

At least duplicate experiments should be performed with each sample of AMK. Without any prior knowledge of tne critical filtration velocity (CFV), it is advisable to start at a low velocity, such as 0.25 cm/s using tne filter backup discs that give the largest effective filter area (i.e., 1D = 0.714 cm. 0.4 cm^2 filter area) and increase the velocity by 0.25 cm/s until the pressure starts to increase with time. If no pressure increase can be found, change the filter backup discs to the next smallest size, and again start the pump at a low velocity. Increase as before until the pressure starts to increase with time. Repeat this procedure until the proper size filter backup discs are found that will cause a pressure rise with time within the flow rate of the pump. This procedure works well for paper filters and relatively fine metal screens (i.e., 16-18 um or less); however, for larger metal screen (40 um or greater) it is suggested that one start at a velocity of 1 to 2 cm/s and increase the velocity by proportionately higher increaments.

Generally, the experiment is not terminated after the first point at which the pressure increases with time. Rather, the rate of pressure rise is measured at increasingly higher flow rates until either the maximum pressure of the transducer or the maximum flow rate of the pump is exceeded. These data are then plotted to make a better estimate of the CFV. A second experiment should be conducted to more accurately estimate the CFV. In this second experiment, a new

filter must be installed and the lines purged of air as described in item 3.
Measurements are then made near the estimated CFV using smaller increments than
were used in the first test.

2.6.2.4 Results and Discussion.

The pump filtration test is able to determine the filtration characteristics of a
degraded AMK sample by measuring the critical filtration velocity. Using the
critical filtration velocity to measure the degradation level of an AMK sample
appears to be a more sensitive test method than the filter ratio test at low
filter ratio values. One would expect that a sample of highly degraded AMK with
a filter ratio of 1.2 would exhibit filtration characteristics similar to Jet A
when, in fact, significantly different results were found when the sample was
tested with the pump filtration test (reference 8). Figure 2.17 shows the pump
filtration data for Jet A and the degraded AMK (FR = 1.2) which showed definite
evidence of filter plugging. This test can easily distinguish between degraded
AMK samples with different degradation levels as shown in figure 2.18 which shows
AMK samples degraded at different specific degrader powers.

The FAA determined, using the FAA pump filtration apparatus, that the critical
flow velocity could also be found for undegraded AMK. The critical flow velocity
of an AMK sample appears to be related to flow rate, effective filter area, and
filter type for all degraded and undegraded AMK samples by finding the right
combination of these variables. For example, the critical flow velocity of a
highly degraded AMK sample was 1.2 cm/sec using a 16 to 18 micron metal screen
with an effective filter area of 0.1 cm^2, and the critical flow velocity of an
undegraded AMK sample was 0.34 cm/sec using a 40 micron metal screen with an
effective filter area of 1.28 cm^2.

2.7 GEL PERMEATION CHROMATOGRAPHY.

2.7.1 Background and Theory.

The antimisting fuel of this Program consists of Jet A aircraft turbine fuel that
is mixed with a low concentration of FM-9 polymer. FM-9 polymer is a high
molecular weight polymer, or in other terms, a large molecular size polymer. In
order for FM-9 antimisting fuel to be compatible with fuel systems components of
a turbine engine, the AMK must be degraded before it is introduced into the
engine fuel system. The process of degrading AMK takes place by passing the AMK
through a mechanical device that produces high shear forces. AMK has been
degraded using a high-speed centrifugal fuel pump, a gear type fuel pump, and by
passing AMK through a needle valve at high pressure. On a molecular scale, the
process of degrading AMK takes place by shearing or breaking the large molecular
size FM-9 polymer molecules into molecules of much smaller molecular size. The
standard laboratory characterization tests, such as the filter ratio and cup
tests lack the sensitivity needed to adequately characterize highly degraded AMK
fuel samples with filter ratios less than 1.5. Gel Permeation Chromatography
(GPC) measurements were made on highly degraded AMK samples in an attempt to
obtain a test method that would give a more sensitive characterization of highly
degraded AMK.
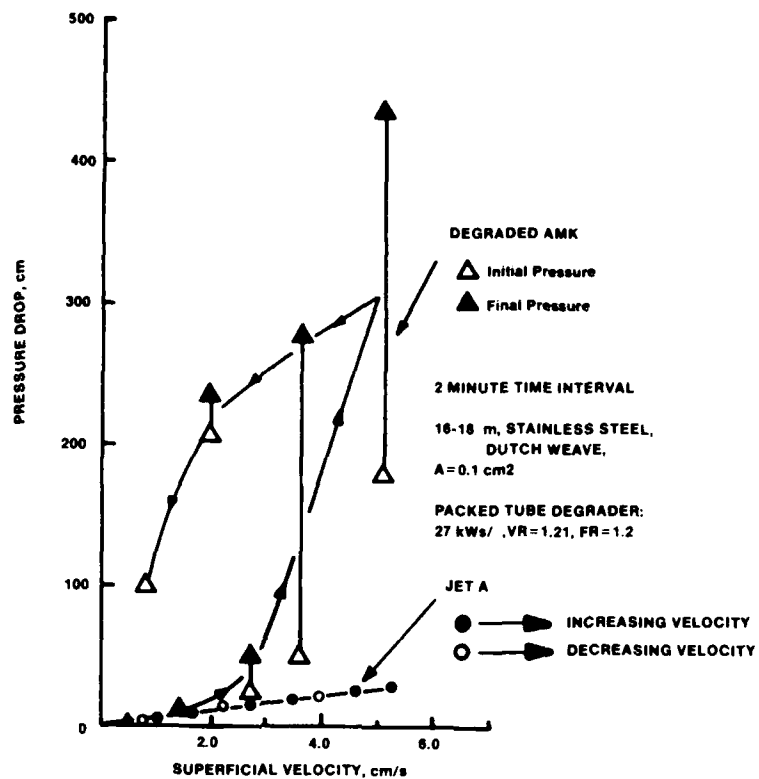
FIGURE 2.17 EFFECT OF SUPERFICIAL VELOCITY ON PRESSURE DROP
ACROSS A FILTER WITH JET A AND DEGRADED AMK
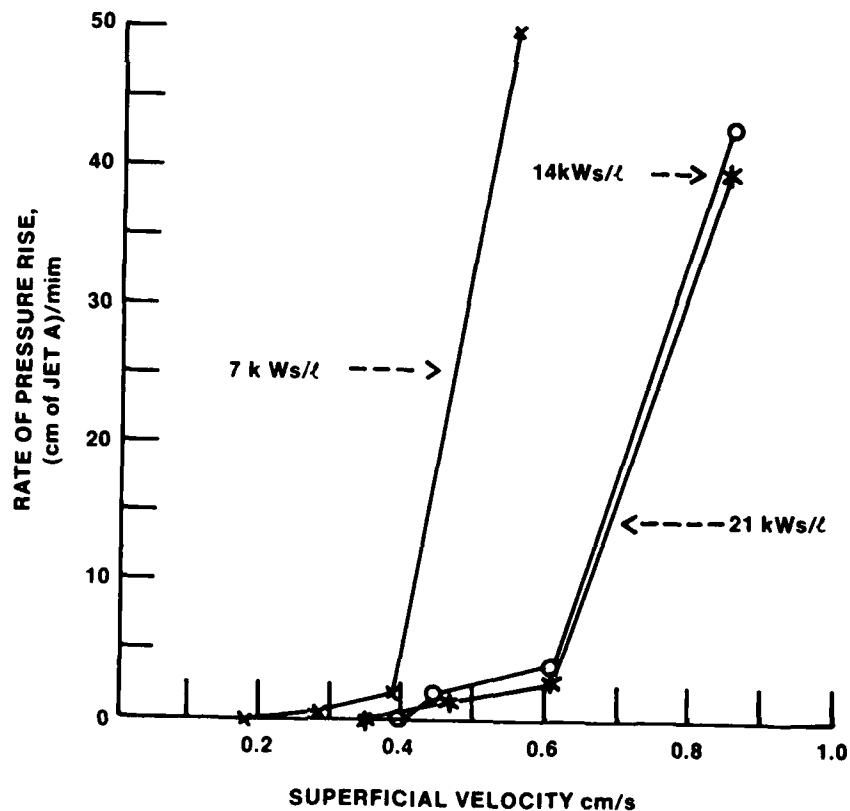
FIGURE 2.18 PUMP FILTRATION DATA FOR AMK DEGRADED AT DIFFERENT
SPECIFIC POWERS (22-24°C)

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GPC, also known as Size Exclusion Chromatography, is a test method that is used to separate the molecules of polymer solutions on the basis of molecular size. GPC is a convenient test method for measuring molecular weight distributions and average molecular weights, provided there are polymer standards available for calibrating the equipment's packed columns. The separation process of GPC is based on entropic interactions with a packed column. Therefore, the retention of the molecules is dependent on the molecular size of the polymer sample and the physical characteristics of the equipment's packed column.

2.7.2.1 Apparatus.

The Perkin-Elmer Series 2 Liquid Chromatography system used by the FAA for GPC measurements is pictured in figure 2.19. The molecular size distribution of a polymer solution is obtained by passing a sample through the packed GPC column which separates the molecules by molecular size. The column is packed with polystrene gel of known geometry and pore size. The largest molecules of the polymer solution tend to flow through the packed column and emerge first because they are too large to permeate the pores of the packing. When the polymer molecules approach the pore size of the packing, they start to diffuse into the internal structure of the packing and elute at later elution times. The smallest molecules which can freely penetrate all of the available pore volume elute last from the column. A diagram of this process is shown in figure 2.20. It is important that the GPC columns are chosen with a pore size range approximately equal to the range of the molecule sizes in the polymer solution.

The GPC apparatus generates a chromatogram that gives the complete molecular size distribution of the sample based on elution volume versus time. The peak elution volume is indicative of the average molecular size of the sample polymer solution (reference 2). Using the information obtained during the GPC process, there are four molecular averages that can be determined. The number-average molecular weight ($\overline{Mn}$), weight-average molecular weight ($\overline{Mw}$), Z-average molecular weight ($\overline{Mz}$), and polydispersity ($\overline{Mw}/\overline{Mn}$) are defined by the following equations.

$$\overline{Mn} = \frac{NiMi}{Ni} = \frac{Wi}{Wi/Mi}$$

The number-average results from a process in which every molecule is counted in the same way, regardless of mass. This average is obtained by dividing the mass of the polymer sample by the total number of chains present. Wi and Ni are the weight and number of molecules of molecular weight, Mi, and i is the incrementing index over all molecular weights present.

$$\overline{Mw} = \frac{NiMi^2}{NiMi} = \frac{WiMi}{Wi}$$

The weight-average results from a process in which each molecule contributes to Mw in proportion to the share of its mass.

$$\overline{Mz} = \frac{\Sigma\ NiMi^3}{\Sigma\ NiMi^2}$$

The Z-average is determined from the refractive index difference between the polymeric solution and a solvent.

$$\frac{\overline{Mw}}{\overline{Mn}}$$

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SEC-2 Environmental Chamber

Series 2 Liquid Chromatograph

LDC Refractomonitor Control Unit

LC-100 Column Oven

LDC Refractomonitor Optical Unit

LC-15 UV Detector

Cole Parmer Dual Channel Chart Recorder
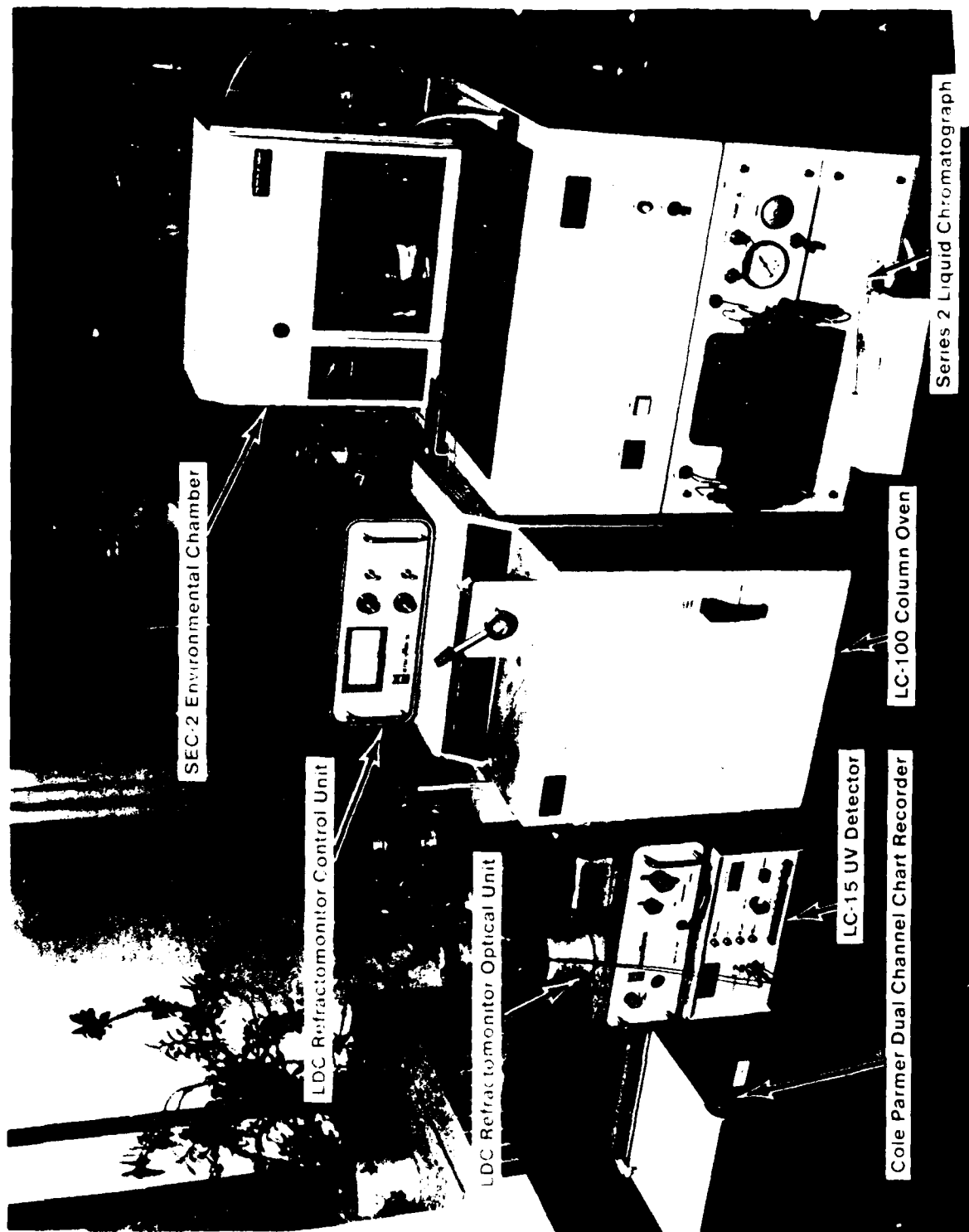
FIGURE 2.19 GEL PERMEATION CHROMATOGRAPH

84-2294
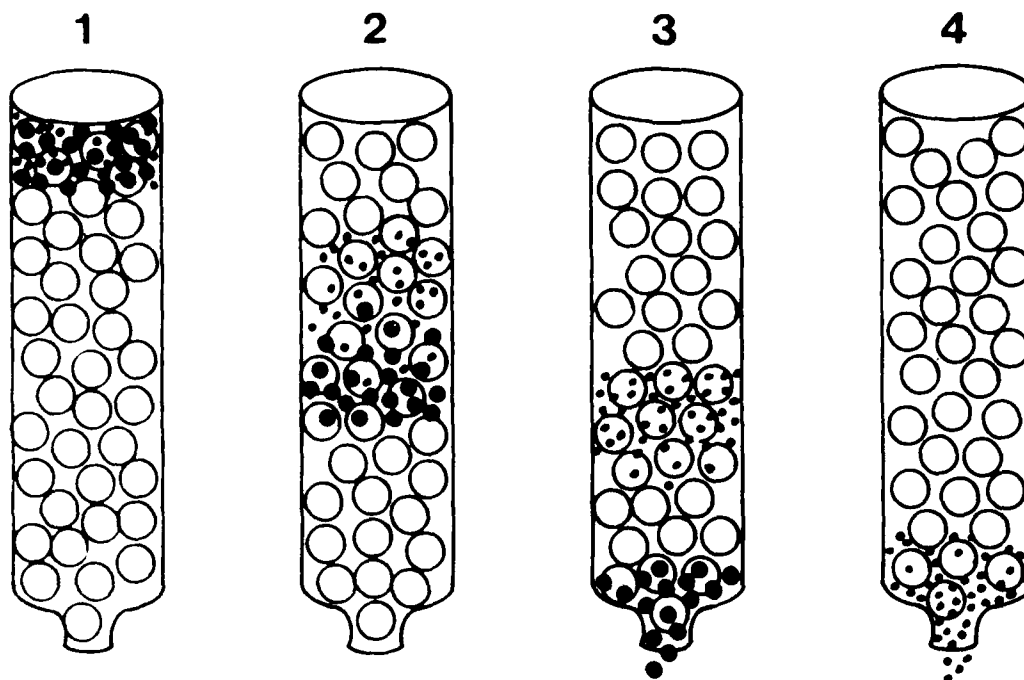
1. Sample ejected on column
2. Size separation
3. Large solutes eluted
4. Small solutes eluted

86-23

FIGURE 2.13. SEPARATION PROCESS IN SIZE EXCLUSION CHROMATOGRAPHY

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The polydispersity is determined by measuring the breadth of the polymer molecular weight distribution. The Perkin-Elmer Sigma 10B data processing unit calculates a calibration curve for the packed column based on the retention times of the polystrene standards and then produces a data table that includes the above parameters, a normalized weight distribution plot and a chromatogram for the test sample, and all this is done automatically by the software provided with the unit (reference 9).

2.7.2.3 Equipment Specifications.

2.7.2.3.1 FAA GPC.

a. Gel Permeation Chromatography Apparatus, Perkin-Elmer Series 2 Liquid Chromatograpny System, which consists of two Series 2 Liquid Chromatograph LC-100 Column Ovens, one LC-15 UV Detector, one LDC Refractomonitor optical unit and control unit, one Cole Parmer dual channel chart recorder, Model 8378-20, and one SEC-2 Environmental Chamber. This system is interfaced with a Perkin-Elmer Sigma 10B data processing unit.

b. Solvent, Tetrahydrofuran, (THF).

c. Columns, Shodex A 80 mls, a short column (8 mm ID x 250 mml) packed with four different gels for a molecular weight range of 1,000 to 50,000 MW, a Shodex A 805/S for a molecular weight range of 100,000 to 50,000 MW, and a Shodex A 805/S for a range of 10,000 to 5,000,000 MW.

d. Calibration standard, polystyrene base, polymers.

2.7.2.3.2 JPL GPC.

a. Gel Permeation Chromatography Apparatus, A. Waters Associates, Model 6000, high performance liquid chromatograph.

b. Solvent, Tetrahydrofuran.

c. Columns, four styragel columns 10^6, 10^4, 10^3 and 500 A molecular weight.

d. Calibration standard, commercial monodisperse polystyrene fractions.

2.7.2.3.3 SWRI GPC.

a. Gel Permeation Chromatograph Apparatus, Waters, ALC 100 Unit, equipped with differential refractiometer and UV detectors.

b. Solvent, Tetrahydrofuran.

c. Columns, four 1-foot microstyrogel columns (10^6, two 10^5, and 10^4 A molecular weight.

d. Calibration Standard, high molecular weight polystyrene, 3.8×10^6 molecular weight.

2.7.2.4 Procedure (Perkin-Elmer Series 2 Liquid Chromatography System)

1. Assemble the GPC apparatus that is shown in figure 2.19. Follow all manufacturers instructions and precautions that are given in the operation manual that is supplied with the apparatus.

2. Turn on the power to all units of the GPC apparatus.

3. Fill the solvent bottle with tetrahydrofuran and purge with helium for 3 minutes.

4. Open drain valve on Series 2 pump module.

5. Vent the solvent lines with 30 ml THF.

6. Close drain valve.

7. Turn on pump, set speed to 3.0 ml per minute.

8. Check visual UV absorbance readout and set to zero.

9. Turn off pump. Connect the analytical column.

10. Set pump speed to 1.0 ml.

11. Allow THF to flow through the column for 20 minutes.

12. Check UV absorbance, zero if possible.

13. Set up the Sigma 1 data processing unit for data collection.

14. Prepare the column calibration standard, dilute the stock polystyrene standard with THF to a 10 to 1 ratio. Filter the solution using the Perkin-Elmer filtration kit for organic sample preparation. Follow the instructions supplied with the kit.

15. Inject the polystyrene calibration standard into sample loop through a Pheodyre 7125 injector with a 10-microliter sample loop. (This is done to obtain a calibration curve for the packed column relative to a polymer of known molecular weight).

16. Activate Sigma.

17. Obtain chromatogram.

18. Push data stop on Sigma.

19. Prepare the AMK sample according to step 14.

20. Flush sample loop per steps 10 through 12.

21. Set up the Sigma 10B data processing unit per step 13.

22. Inject the diluted AMK test sample into the sample loop per step 15.

23. Activate Sigma.

24. Obtain the chromatogram; table of molecular weights based on time or volume of solvent eluted; an average number, weight, and Z molecular weight; and a log molecular weight versus weight distribution plot from the Sigma 10B data processing unit for the AMK test sample.

25. Push data stop on Sigma.

26. Allow Jet A peak to elute on recorder.

2.7.2.4 Results and Discussion.

Based on a limited amount of testing conducted by the FAA, JPL, and SWRI, gel permeation chromatography appears to be a more sensitive test method than filter ratio or the cup test for characterizing highly degraded AMK. Table 2 shows that the weight average molecular weight determined by the GPC test can indicate the degree of degradation of AMK samples degraded in a blender for various lengths of time. Even though the degradation times are relatively close in some cases, the weight average molecular weight results can distinguish between them.

Gel Permeation Chromatography is a convenient test method for measuring molecular weight distributions and average molecular weight provided that polymer standards are available for calibration of the packed columns. Since the calibration standards used for antimisting fuel testing are polystrene standards, the values obtained for the average molecular weights of the AMK sample are only relative numbers based on the calibration standard (reference 10). For the numbers to be absolute, an FM-9 polymer calibration standard would have to be used.

Mannheimer, of SWRI, reported GPC results in terms of the peak elution volume (PEV) which approximates the average molecular size of the polymer sample. Testing conducted at SWRI determined that there was a correlation between the peak molecular weight and the filter ratio of degraded AMK samples. It was also determined that the results produced from gel permeation chromatography may not indicate the filter plugging tendencies that are sometimes exhibited by degraded AMK samples that were degraded immediately after inline blending. This tendency appears to be caused by a small quantity of poorly solubilized FM-9 polymer which can readily clog a fine filter such as the filter in the filter ratio test (reference 2).

2.8 DIE SWELL TEST.

2.8.1 Background and Theory.

Researchers determined that FM-9 antimisting fuel additive would have to be inline blended into the Jet A fuel at the aircraft's refueling point for the concept of the use of antimisting fuel in the civil aviation fleet to be feasible. With the successful development of the inline blending process for antimisting fuel, it became apparent that there would also be a need for a single real-time quality control test that could access the quality of the antimisting fuel in the fuel tanks of an aircraft before the aircraft proceeded for takeoff. The die swell test was developed for antimisting fuel by SWRI and further modified by the FAA as a real-time quality control test for AMK.

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TABLE ... RELATIVE MOLECULAR WEIGHTS OF BLENDER AND ULTRASONIC SAMPLES BY GEL PERMEATION CHROMATOGRAPHY

Degradation Time in Min	Blender			Ultrasonic		
	$\overline{M}_n$	$\overline{M}_w$	$\overline{M}_w/\overline{M}_n$	$\overline{M}_n$	$\overline{M}_w$	$\overline{M}_w/\overline{M}_n$
1	744,000	1,150,000	1.55	521,000	886,000	1.70
2	672,000	1,130,000	1.68	444,000	848,000	1.91
3	564,000	1,050,000	1.86	336,000	768,000	2.29
5	612,000	980,000	1.60	363,000	733,000	2.02
10	587,000	918,000	1.56	236,000	530,000	2.25
20	579,000	930,000	1.61	193,000	388,000	2.01

$\overline{M}_n = \sum NM / \sum N$

$\overline{M}_w = \sum NM^2 / \sum NM$

N = number of molecules

$\overline{M}_n$ = number average molecular weight

$\overline{M}_w$ = weight average molecular weight

M = molecular weight

Capillary tube experiments conducted by Mannheimer of SWRI showed the viscoelastic behavior of antimisting fuel when the critical shear rate was exceeded. This viscoelastic behavior became apparent when the flow stream that was discharged from the capillary tube exhibited swelling (increase in diameter) caused by large normal stresses that are reported to be 10 to 100 times greater than the shear stresses (reference 11).

The antimisting phenomenon that is characteristic of dilute polymer solutions appears to be a result of the high extensional viscosity of the solution. Since attempts to measure the extensional viscosity of antimisting fuel were not successful, die swell measurements were conducted to estimate the normal stresses developed by AMK at high shear rates. By the use of an optical measurement device, die swell (diameter) measurements can be made of the flowstream issuing from a capillary tube. The amount of swelling (increase in diameter) is directly related to the polymer concentration in the test solution and is proportional to the normal stresses that are developed.

Since the development of large normal stresses occurs at high shear rates, the amount of swelling is, therefore, related to the shear rate developed. The die swell test apparatus can vary the shear rate by increasing or decreasing the speed of the hydraulic pump to change the flow rate of the AMK test sample through the capillary tube. The apparent shear rate can be varied from 400 sec^{-1} to 17,435 sec^{-1} by changing the setting on the hydraulic pump. To standardize the die swell test method, the following pump settings were chosen for testing.

PUMP SETTING	SHEAR RATE (SEC^{-1})
14	4409
16	5311
18	6279
20	7340
22	8450

All of the pump settings shown above produce shear rates above the critical shear rate of FM-9 antimisting fuel, which is the point where AMK begins to exhibit non-Newtonian flow behavior.

2.8.2 Experimental.

2.8.2.1 Apparatus.

The prototype die swell apparatus that was constructed by SWRI, and used for all die swell measurements during the AMK program, is shown in figure 2.21. A schematic of the major components of the die swell test apparatus is given in figure 2.22. The main components of the die swell test apparatus are the hydraulic cylinders, the capillary tube, and the optical measurement sensor. There are two hydraulic cylinders connected in tandem. One hydraulic cylinder is controlled by a Zenith precision metering pump system. Diverting the direction of the flow from the metering pump to opposite ends of the hydraulic cylinder causes the cylinder to operate in forward and reverse direction. This causes the tandem cylinder to function as a pump. In one direction, the antimisting fuel test sample is loaded into the hydraulic cylinder from the sample reservoir, and in the opposite direction, the AMK is pushed out of the hydraulic cylinder where it is discharged through the capillary tube.

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FIGURE 2.21 DIE SWELL RHEOMETER

DIE SWELL

HYDRAULIC FLUID

AMK
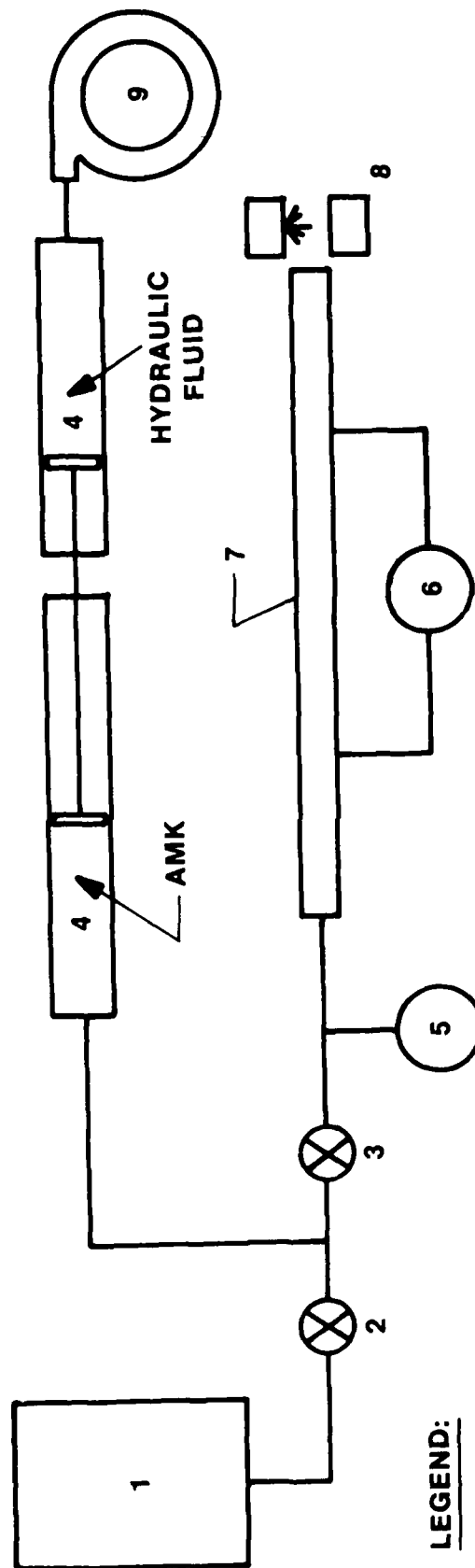
LEGEND:

1) RESERVOIR
2) VALVE FOR LOADING FUEL
3) VALVE FOR CAPILLARY TUBE
4) HYDRAULIC CYLINDER
5) PRESSURE GUAGE, TOTAL PRESSURE DROP
6) PRESSURE GUAGE, PRESSURE DROP FROM L/R OF 50 TO L/R OF 150
7) CAPILLARY TUBE, L/R 200
8) OPTICAL SENSOR
9) VARIABLE SPEED HYDRAULIC PUMP

FIGURE 2.22 SCHEMATIC OF THE DIE SWELL APPARATUS

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Typically, when AMK is pumped by a mechanical pump that shears the fuel, the AMK becomes degraded to some extent. The use of the hydraulic cylinder as a pump keeps the level of unintentional degradation of the AMK sample to a minimum. The test fuel sample is loaded into the hydraulic pump cylinder at a low metering pump setting also to minimize any unintentional degradation of the AMK sample.

After the fuel is discharged from the hydraulic cylinder pump, the fuel passes through the capillary tube at a fixed shear rate determined by hydraulic metering pump setting. The shear rate can be changed by changing the pump setting (flow rate of the hydraulic metering pump. This affects the change in the speed at which the piston of the hydraulic cylinder pump moves and the flow rate of the AMK changes accordingly. When the flow rate of the AMK is increased, there is a corresponding increase in pressure at the inlet of the capillary tube which causes the shear rate to be increased, due to the increase in flow through the capillary tube of fixed diameter.

When the antimisting fuel is discharged from the capillary tube, the die swell measurement is made. The diameter of the flow stream is measured by an optical sensor close to the end of the capillary tube and the output is recorded on a strip chart recorder. The pressure drop across the capillary tube is also measured using a pressure transducer located at the inlet of the capillary tube. The pressure drop measurement is made at the same time the die swell measurement is made and is also recorded on the strip chart recorder. Pressure drop and die swell measurements are made at five different shear rates which are indicated by the specified setting of the hydraulic metering pump. A typical strip chart recording of a complete die swell test for FM-9 antimisting fuel is shown in Figure 2.23, along with with a legend for the data on the strip chart recording.

From the data recorded on the strip chart during the test procedure, a determination of the effective concentration of the antimisting fuel test sample can be made using the quality control curves generated during the initial development of the die swell apparatus. The complete set of quality control curves consist of two groups of curves separated according to the age (time after blending) of the AMK. The set consists of a 5-minute-old AMK curve group and a 20-minute-old AMK curve group. Each group consists of seven curves: five curves show the pressure drop versus concentration at each specified shear rate setting, one curve shows the jet diameter versus concentration at a shear rate of 4409 sec^{-1}, and one curve shows the delta divisions versus concentration. Figures 2.24, 2.25, and 2.26 show the complete set of curves for 5-minute-old FM-9 antimisting fuel.

2.2.1 Equipment Specifications.

1. Metering Pump, Zenith Precision Metering System, Model ZM, manufactured by Zenith Products Company, West Newton, Massachusetts.

2. Hydraulic Distribution Valve, manufactured by Victor Fluid Power, Inc., Franklin Park, Illinois.

3. Hydraulic Cylinder, 2.5-inch bore x 13-inch stroke, Model 71 J, manufactured by Victor Fluid Power, Hammond, Indiana (3 required).

4. Optical Sensor with Analog Voltage Output, Model 844800, manufactured by C. W. Brabender Instruments, Inc., Hackensak, New Jersey.

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Pump Setting	Shear Rate (sec.⁻¹)
14	4409
16	5311
18	6279
20	7348
22	8450

PRESSURE (DIVISIONS)

JET DIAMETER - (mm)

PUMP SETTING
:14
:16
:18
:20
:22

JET DIAMETER

PRESSURE

◄— TIME

Strip chart Legend

100

Date: 09/25/84
Slurry: 16-95
Concentration: 0.27%
Fuel Temperature 20.5°C
Age: 20 Minutes
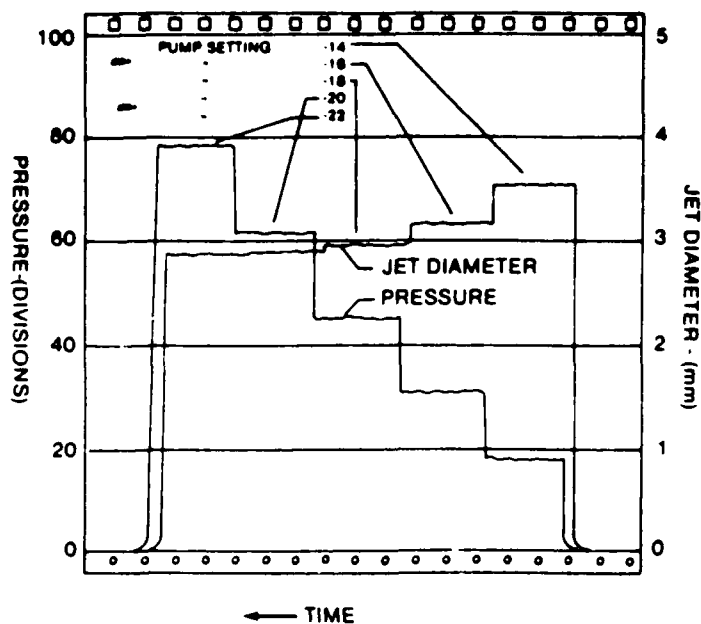
JET DIAMETER
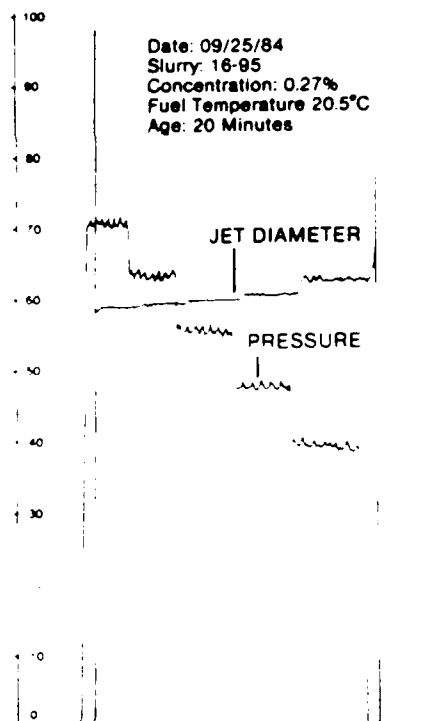
PRESSURE

**Reproduction of a Strip
Chart Recording**

FIGURE 2.23. STRIP CHART LEGEND AND A REPRODUCTION
OF A STRIP CHART RECORDING

47
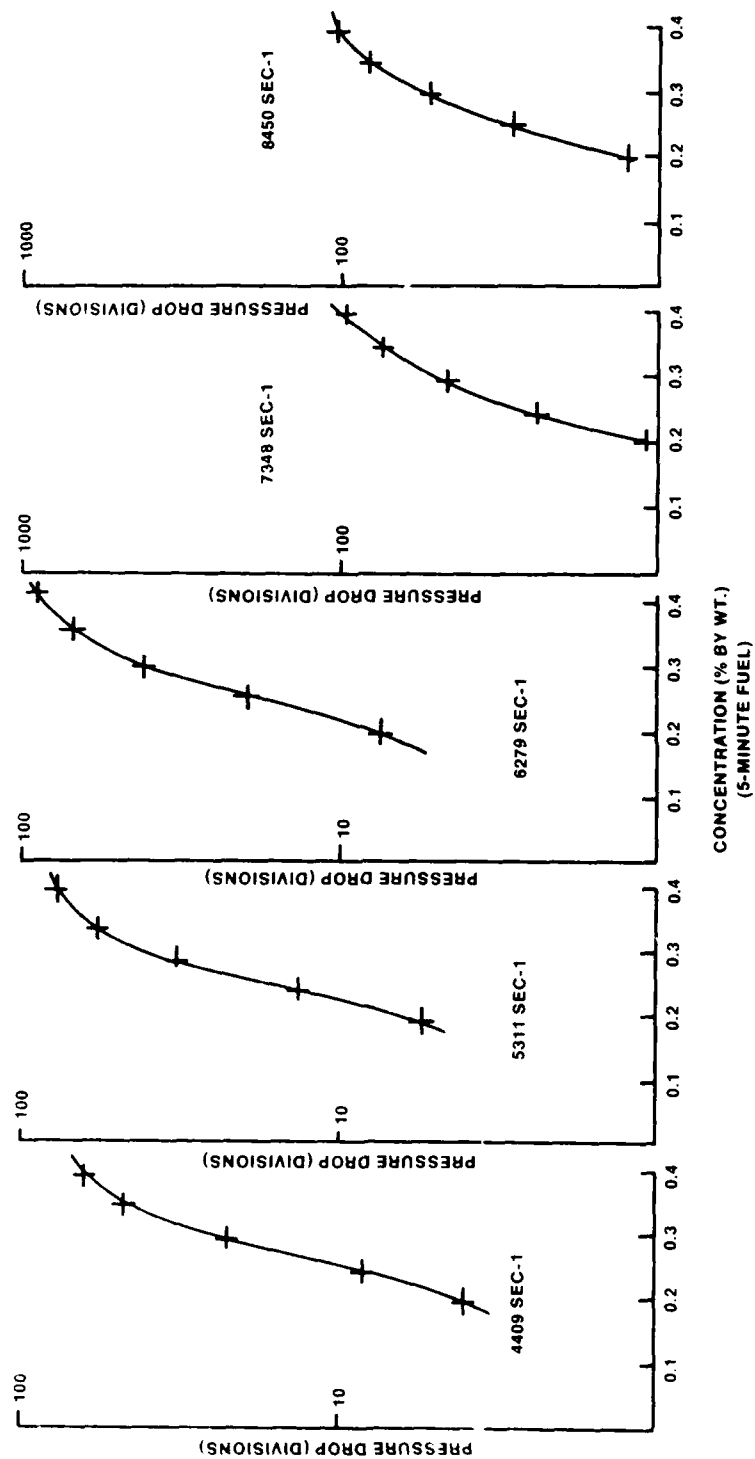
FIGURE 2.24 PRESSURE DROP VERSUS CONCENTRATION FOR FIVE SHEAR RATES

48
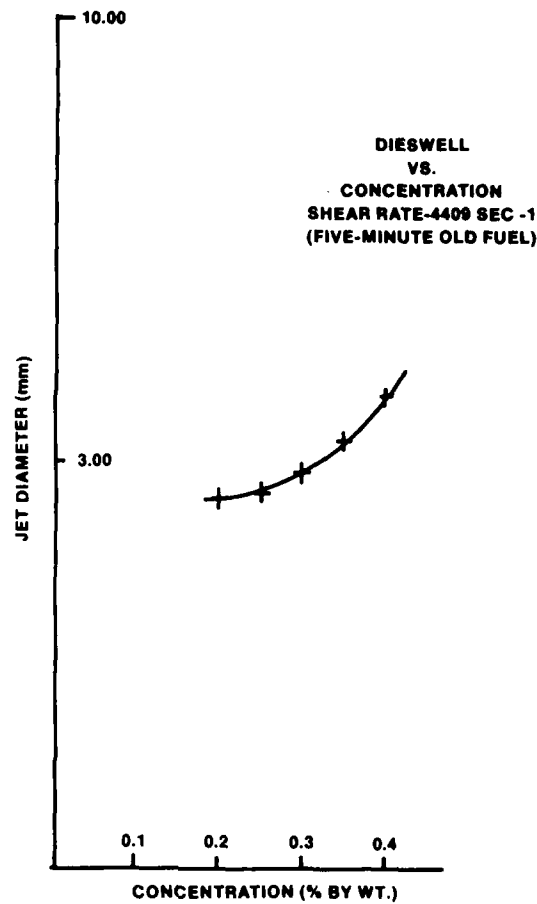
FIGURE 2.25   JET DIAMETER VERSUS CONCENTRATION
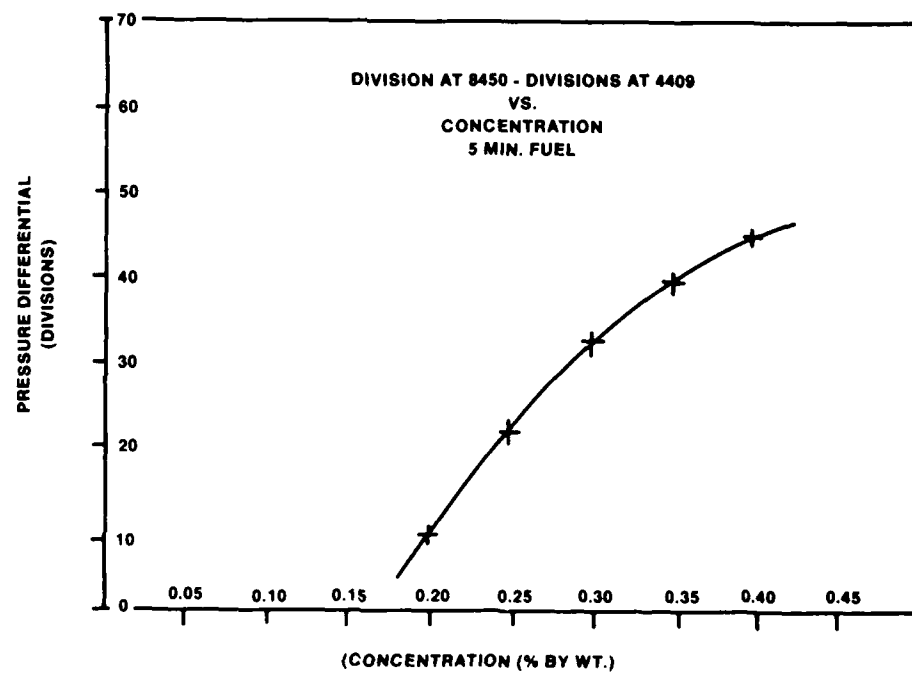
FIGURE 2.26   PRESSURE DIFFERENTIAL VERSUS CONCENTRATION

5. Strip Chart Recorder, Model 242, manufactured by the Soltec Corporation, Sun Valley, California.

6. Stainless Steel Capillary Tube, internal diameter 2.95 mm, length 281.725 mm (L/R = 191).

7. 25 psi Pressure Transducer.

8. 10 psi Pressure Transducer.

9. 3/8-inch Swagelock Ball Valve.

10. 1/4-inch Swagelock Ball Valve.

11. Suitable reservoir for test fuel and hydraulic fluid.

12. Stainless steel tubing, 1/2 inch, 3/8 inch, 1/4 inch, and 1/8 inch outside diameter.

13. Miscellaneous Swagelock Fittings for tubing, specified in Number 12.

2.8.2.3 Procedure.

1. Remove the cover from the unit. Place a pail under the drain.

2. Put the hydraulic reservoir in place and fill it with filtered hydraulic fluid. Cover the reservoir with aluminum foil.

3. Put the AMK reservoir in place and secure it. Keep the reservoir covered as much as possible.

4. Place the sensor assembly in the track.

5. Secure all electrical connections.

6. Adjust the zero reading.

7. If the unit cannot be zeroed, it is necessary to clean the glass surfaces of the sensor by loosening the Allen screws on the right hand face of the sensor. Use an alcohol swab to clean the surfaces. Reassemble the sensor using caution when securing the Allen screws, since the body is made of aluminum.

8. Put the span unit in the sensor. Keep the span unit in the green area. Adjust the reading to 5.99 mm.

9. Repeat steps 6 and 8 as necessary.

10. Put the capillary tube in place and carefully align the sensor. Lock the sensor in place (large Allen screw on the bottom surface). Gently secure the cat's eye clamp. There should be enough adjustment to either cover the tube with sensor unit or have the tube completely uncovered

11. Check the sensor calibration by running the sensor over the capillary tube. At least 1 cm of tube should be in the sensor. It should read 3.275 mm ±0.005 mm.

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12. Make sure the tube is 1 mm from the sensor. To do this, turn the adjustment knob so that the unit just begins to sense a change from zero. Back off 1 small division (i.e., 1 mm).

13. Pour about 1 liter of the test sample in the reservoir. Open both valves. Allow enough sample to discharge so that the air is purged from the lines. Close the valve to the capillary tube.

14. Set the pump speed to 10. Start the pump in Neutral (Neutral is the center position). Load the cylinder by placing the pump in reverse (move the handle to the right). After the mark appears in the sight glass, put the pump in Neutral. The pump may be left in Neutral for a limited period of time. Do not allow the reservoir to run dry.

15. Select flow rates and shear rates from the calibration chart. Set the pump speed to the starting position. If the tube is horizontal, start with a shear rate greater than 4000 sec^{-1} so that the jet does not droop below the green area.

16. Check voltage settings on the die swell unit and the strip chart.

17. Record all the data on the strip chart including the date, time, fuel type, and voltage/scale settings.

18. Close the valve to the reservoir and open the valve to the capillary tube.

19. Start the run by moving the pump control lever to the left. After making the reading, the pump speed may be changed as the pump is moving. Do not exceed the cylinder limitations. This may take as little as 15 seconds at the higher flow rates. Record the die swell indication.

20. To reload the system, drop the pump speed to 10. Close the capillary tube valve and open the reservoir valve. Do not draw air into the system.

21. Repeat steps 10 through 20 for all the points selected in step 15.

22. At the end of the run, pump all fuel from the cylinder by going full forward. Open the reservoir valve and allow the sample to drain out.

23. Remove the reservoir and capillary tube. Cap the fittings and clean the tube and reservoir.

24. Turn off all power. If the unit will be idle for an extended period of time, remove the hydraulic reservoir and cap the line and secure cover.

25. Reduce the data according to the following procedure. Some key parameters are listed below.

ρ AMK $= 0.808$ gm/cm^3 @ 15°C

$\overline{\text{D}}$ $= 2.90$ mm (used for 8V/D cal.)

L/R $= 191$

D $= 2.94$ mm (used for die swell calculations)

8/VD $= 6.68Q$ where Q is ml/min

$\qquad$ 8V/D is sec^{-1}

$\tau 12$ $= 180.3$ ΔP where ΔP is psig

$\qquad$ 12 is dyne/cm^2

n = Slope of the shear stress versus corrected shear rate curve, Pa sec

n' = Slope of the shear stress versus uncorrected shear rate curve, Pa sec

$\qquad$ (a) Determine the total pressure drop based on the strip chart reading.

$\qquad$ (b) Determine Q based on the calibration sheet.

$\qquad$ (c) Calculate 8V/D by multiplying Q by 6.68. The units are sec^{-1}.

$\qquad$ (d) Calculate τ_{12a}, the apparent wall shear stress, by multiplying the total pressure drop by 180.3. The units are dynes/cm^2. Divide by 10 to get Pa.

$\qquad$ (e) Read D_j off of the strip chart or by recording the indicator readings. If the indicator readings are recorded, cross check with the strip chart.

$\qquad$ (f) Calculate D_j/D by dividing D_j by 2.94 mm. The result is dimensionless.

$\qquad$ (g) Plot D_j/D verses 8V/D on semi-log paper for comparison purposes.

$\qquad$ (h) Plot $\tau 12a$ versus 8V/D on a log log scale. This shows the linear area for calculating n'.

$\qquad$ (i) If available, determine the corrected pressure drop of the different shear rates by plotting the pressure drop for a number of tube lengths (L/R = 300, 300, and 400) for a given radius and shear rate. Determine the end correction by plotting the y-intercept of the data. The corrected pressure drop is:

$\qquad$ $P_c = \Delta P - P_e$

where P_c is the corrected pressure

$\qquad$ P_e is the end correction

Correct P_c for inertial effects by subtracting 1.1 ρV^2, $P = P_c - 1.1 \rho V^2$

(j) An alternate is to use the wall shear stress technique developed at the Technical Center (reference 1). In this case,

P = the pressure measured across the two taps (psid) and
τ_{12} = 180.3 P_2 (dyne cm^2), for L/R = 191, r = 2.90

Generally, $\tau_{12} = A\dfrac{P}{w}$

where A_w is the area of the tube wall.

(k) Plot $\tau 12$ versus 8V/D on log log paper.

$$n' = \frac{d \log \tau 12}{d \log 8V/D}$$

An easy way to calculate n' is to do a linear regression over the linear region determined above. The slope is n'.

1) Calculate $\dot\gamma_R$ the corrected shear rate:

$$\dot\gamma_R = \left(\frac{3 + 1/n'}{4} \right) \left(\frac{8V}{D} \right)$$

(m) Calculate n

$$n = \frac{d \log \tau_{12}}{d\log \dot\gamma_R}$$

n) Calculate $(v_{11} - v_{22})\big|_{\dot\gamma}$ the normal stress for a given shear rate using:

$$(v_{11} - v_{12})\big|_{\dot\gamma} = \frac{\rho D^2 (8V/D)^2}{64n'} \left\{ (n' + 1)\left(\frac{3n + 1}{2n + 1} \right) - \left(\frac{D}{D_j} \right)^2 \left(n' + 1 + \frac{d \log D/D_j}{d \log 8V/D} \right) \right\}$$

(o) Plot τ_{12} and $v_{11} - v_{22}$ on a log log scale versus R
(p) The intrinsic viscosity for a given shear rate is:

$$\eta = \frac{R\ P/2L}{\dot\gamma_R} \quad \text{or} \quad \frac{D\ P/4L}{\dot\gamma_R}$$

53

2.8.2.4 Results and Discussion.

The initial testing of the die swell rheometer was conducted with inline blended AMK that was blended from the same batch of slurry. All of the slurry contained FM-9 polymer from the same polymer lot. The initial testing led to the conclusion that the actual concentration of an AMK sample could be determined using the die swell test procedure. However, when AMK samples blended from slurries that contained FM-9 polymer from different polymer lots were tested, the die swell rheometer did not determine the actual concentration of the AMK samples. The antimisting fuel samples produced from different slurries with different FM-9 polymer lots produced varying die swell measurements even though the AMK samples had the same polymer concentration. Further testing revealed that even though the AMK samples had the same polymer concentration, the samples had slightly different antimisting properties and flammability protection as determined by filter ratio, cup test, and wing spillage test results. This accounted for the difference in the concentration determined by the die swell rheometer.

For the die swell rheometer to be an effective quality control test, it was necessary to find a correlation between the determined concentration measured by the die swell apparatus and the flammability protection provided by the same antimisting fuel. Using the quality control curves that were generated during the initial development of the die swell rheometer using the same batch of slurry from the same polymer lot, researchers determined that an effective concentration could be determined. The effective concentration is relative to the antimisting properties of the initial slurry batch and can be determined by using the following formula (reference 12):

$$\text{EFFECTIVE CONCENTRATION} = \frac{x + y + z}{3}$$

where:

    x = The effective concentration from the Die Swell versus Concentration Curve,

    y = The average effective concentration from the pressure drop versus Concentration Curves, and

    z = The effective concentration from the differential pressure versus Concentration Curve.

A correlation was developed between the effective concentration and the flammability protection provided by the antimisting fuel on the Wing Spillage Facility at the FAA Technical Center. The correlation between the Wing Spillage test results and the effective concentration is shown in figure 2.27.

Die swell test results have also shown that there is a linear relationship between polymer concentration of the AMK sample and the maximum apparent viscosity when corrected for viscoelastic and effects caused by the capillary tube. Since it is very difficult to estimate the end effects, the Technical Center modified the die swell rheometer by installing two pressure taps in the
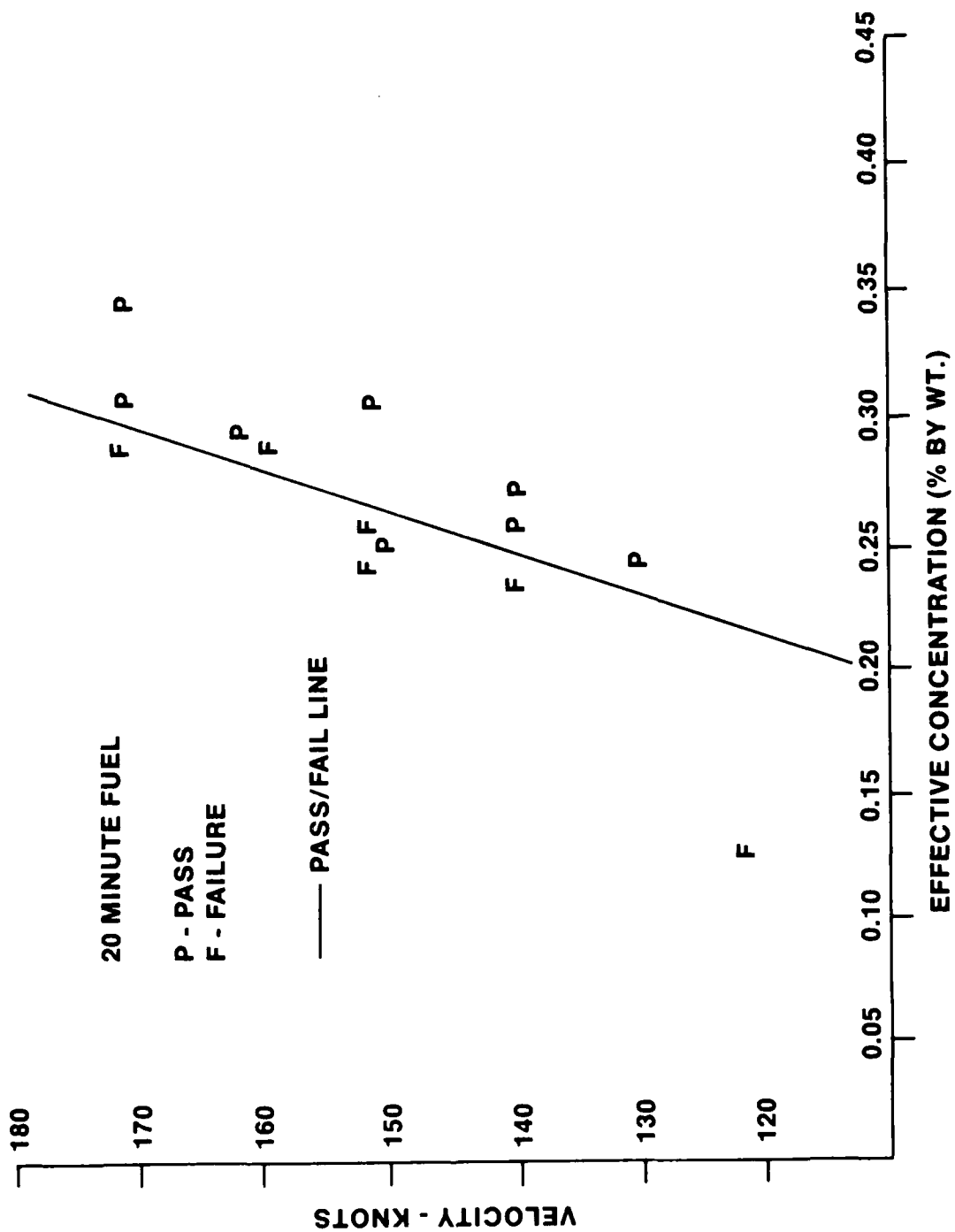
FIGURE 2.27 WING SPILLAGE TEST RESULTS VERSUS EFFECTIVE CONCENTRATION

55

capillary tube which allowed for the measurement of differential pressure in the capillary tube. The shear stress and apparent viscosity could then be determined without the need for end corrections (reference 12), and by changing the positions of the pressure taps in the capillary tube, the effect of residence time at a high shear rate was also investigated.

The die swell rheometer shows the potential of being an effective real-time quality control test that can access the antimisting properties of AMK immediately after inline blending. Using the pressure drop and flow stream diameter measurement obtained from the die swell rheometer, the effective concentration of an antimisting fuel sample can be determined, and there is a linear relationship between the effective concentration and flammability protection of an AMK sample.

2.9 LOW TEMPERATURE GEL FORMATION TEST.

2.9.1 Background and Theory.

Experiments have shown that some cold (-30°C) FM-9 AMK fuels form a stable gel when slightly agitated at low temperatures (reference 10). This gel formation leads to poor low temperature pumpability and excessive fuel holdup in fuel tanks. The low temperature gel formation test was developed and used by Jet Propulsion Laboratory as a quick and repeatable means of identifying individual AMK samples which exhibit this behavior at low temperatures. Very low temperature AMK fuel is passed through a metal screen to observe any transparent gel that may form or be collected on the top of the screen. The test is based on visual observations of the gel collected on the screen when inline blended AMK fuel is tested.

2.9.2 Experimental.

2.9.2.1 Apparatus.

The low temperature gel formation test apparatus consists of ordinary laboratory equipment that is readily available in most laboratories. A filtering flask is used to hold a measured quantity of an AMK fuel sample. The flask is then inerted, sealed, and cooled. After the fuel has reached -25°C, the sample is then stirred for a fixed period of time. This agitation causes any gel formation which is going to take place. Immediately after stirring, the AMK sample is poured through a metal screen.

2.9.2.2 Equipment Specifications.

1. Filter flask, heavy wall, 1000 ml.

2. 1-hole rubber stopper, No. 8.

3. Thermometer, (-35 to 50°C).

4. Tubing, 3/8 inch I.D.

5. Stopcock, 3/8 inch.

6. CO₂/acetone bath.

7. Dry nitrogen gas.

8. Magnetic stirrer.

9. Stainless steel screen, 4 mesh.

2.9.2.3.

1. Insert the magnetic stirrer and pour 400 ml of an AMK fuel sample into the filtering flask and place the stopper in the top of the flask.

2. Insert the thermometer through the stopper into the flask so that the bulb of the thermometer is immersed in the AMK.

3. Attach one end of the tubing to the flask and the other end to the stopcock. Next, connect the stopcock to the nitrogen gas supply.

4. Inert the head space above the AMK sample with dry nitrogen gas, and then close the stopcock.

5. Disconnect the nitrogen gas supply from the stopcock and immerse the flask halfway into the CO_2/acetone bath at -30C.

6. After the AMK sample reaches $-25^{\circ}C$, turn on the magnetic stirrer.

7. Continue to cool and stir the fuel for 10 minutes.

8. Immediately after the 10-minute period, pour the AMK sample through a 4-mesh stainless steel screen as fast as possible.

9. Make a visual observation of any transparent gel on the top of the screen. Note the relative amount of gel and its behavior with time (warming to ambient temperature).

2.9.2.4 Results and Discussion.

The low temperature gel formation test provides a laboratory scale simulation of the low temperature and agitation that could be encountered in an aircraft fuel tank during actual flight conditions. The results of this test should only be used as an indication of which AMK samples warrant further large-scale evaluation in a test facility.

The variables for this test method are temperature and agitiation. The temperature and agitation imparted on an AMK sample should be held constant and repeated each time the test is done. This allows a direct comparison of the low temperature behavior of different AMK samples. The test is a pass or fail, depending on the collection of gel on top of the screen, because when batch blended 0.3 percent FM-9 AMF fuel is used as the test sample, no gel is collected on the screen (reference 13).

2.10. INERTIAL RHEOMETRIC PUMP TEST.

2.10.1 Background and Theory.

The Inertial Rheometric Pump Test was developed at the FAA Technical Center in order to measure the antimisting properties of antimisting fuel. A U.S. patent was awarded to the test in September 1983 under the title of "Rheological Testing Method."

Jet A fuel is a fluid which exhibits Newtonian flow behavior where the flow is directly proportional to the force applied to the fluid. This test is based on the fact that when Jet A fuel is mixed with a small amount of a high molecular polymer, the resulting antimisting fuel exhibits non-Newtonian flow behavior where the flow of AMK is no longer directly proportional to the force applied. In other words, the flow of AMK remains proportional to the force applied up to a point where the flow will then decrease or increase at a slower rate as more force is applied (reference 14).

The test was designed by using a fluid metering pump to apply the force to the test fuel. The pump is a positive displacement piston pump with a variable piston stroke. By varying the stroke of the piston, the displacement of the pump changes and affects a change in the force applied to the test fuel. This happens because the fluid flow is discharged from the pump through a tube of fixed dimensions (inside diameter and length). The force changes due to the fact that a larger or smaller quantity of fuel is being forced through the tube. The tube dimensions were determined by testing so that the force applied in the range of the pump would be above and below the critical shear rate of the antimisting fuel.

2.10.2 Experimental.

2.10.2.1 Apparatus.

The major component of the Inertial Rheometric Pump test is the FMI lab pump which is a positive displacement piston pump. The delivery volume of the pump can be varied by changing the stroke of the piston in the cylinder. By changing the stroke of the piston, rather than restricting or bypassing the flow, the pump remains a positive displacement pump.

The test is conducted by operating the FMI lab pump to draw the antimisting test fuel from the inlet flask through a measured length of small diameter stainless steel tubing that contains several loops with a specified diameter. The pump then discharges the test fuel to the outlet flask again through a measured length of small diameter tubing that contains several loops with a specified diameter. During the test, a measured amount of antimisting test fuel at the specified test temperature is transferred from the inlet flask to the discharge flask by the FMI lab pump set a specified percentage of the pump's piston stroke. The time required to transfer the test fuel completely from the inlet flask to the discharge flask is measured and recorded. This procedure is then repeated in steps of ten from 0 to 100 percent of the pump's piston stroke. The data is then reduced to yield milliliters of test fuel pumped in 188.4 seconds. This time coincides with the time it takes for the FMI lab pump to transfer 750 ml of Jet A to the discharge flask at a pump stroke setting of 100 percent. This calculation changes the variable measured during the test from the time per unit volume to

the volume per unit time. The data is then plotted on the basis of milliliters pumped per 188.4 seconds versus percent of piston stroke.

2.10.2.2 Equipment Specifications.

    1. FMI lab pump, Model RPG-400, manufactured by Fluid Metering, Inc., Oyster Bay, New York.

    2. Pump inlet tube, 186 cm of 1/8-inch stainless steel tubing with 3 3/4 turns of 3-inch diameter in it as shown in figure 2.28.

    3. Pump discharge tube, 315 cm of 1/8 in stainless steel tubing with 4 turns of 3 inch diameter in it as shown in figure 2.28.

    4. Erlenmeyer flask, 1000 ml, clear, two required.

    5. Timer, capable of reading to a tenth of a second.

    6. Thermometer, accurate to 1 degree Celsius and of applicable range.

    7. Constant temperature water bath.

2.10.2.3 Procedure.

    1. Assemble Inertial Rheometric pump test apparatus as shown in figure 2.28.

    2. Place 7.5 liters of the test fuel sample in a constant temperature water bath set at 22 degrees Celsius +2 degrees Celsius.

    3. Bring the test fuel sample to a temperature of 22 degrees Celsius.

    4. Pour 750 ml of the test fuel gently into the inlet Erlenmeyer flask.

    5. Adjust the FMI lab pump to a setting of 10 percent of the maximum piston stroke of the pump by moving the lever near the base to the 10 percent mark indicated on the base of the pump.

    6. Start the FMI lab pump and simultaneously start the timer.

    7. Measure the time it takes for the FMI lab pump to pump the 750 ml of test fuel at the 10 percent stroke setting, stop the timer when the inlet flask is empty.

    8. Record the time to the nearest tenth of a second.

    9. Repeat steps 4 through 8 for pump stroke settings of 20, 30, 40, 50, 60, 70, 80, 90, and 100 percent.

    10. Reduce the data on the basis of the number of milliliters of test fuel pumped in 188.4 seconds for each pump stroke setting using the following calculation:

$$\text{Milliliters (pumped per 188.4 seconds)} = 750 \text{ ml} \times \frac{188.4 \text{ sec.}}{\text{Test Time sec.}}$$

PUMP DISPLACEMENT AT
DIFFERENT SETTING

10	.6787cm
9	.6108cm
8	.5430cm
7	.4750cm
6	.4072cm
5	.3396cm
4	.2715cm
3	.2036cm
2	.1357cm
1	.0679cm

DISCHARGE SIDE

INLET SIDE
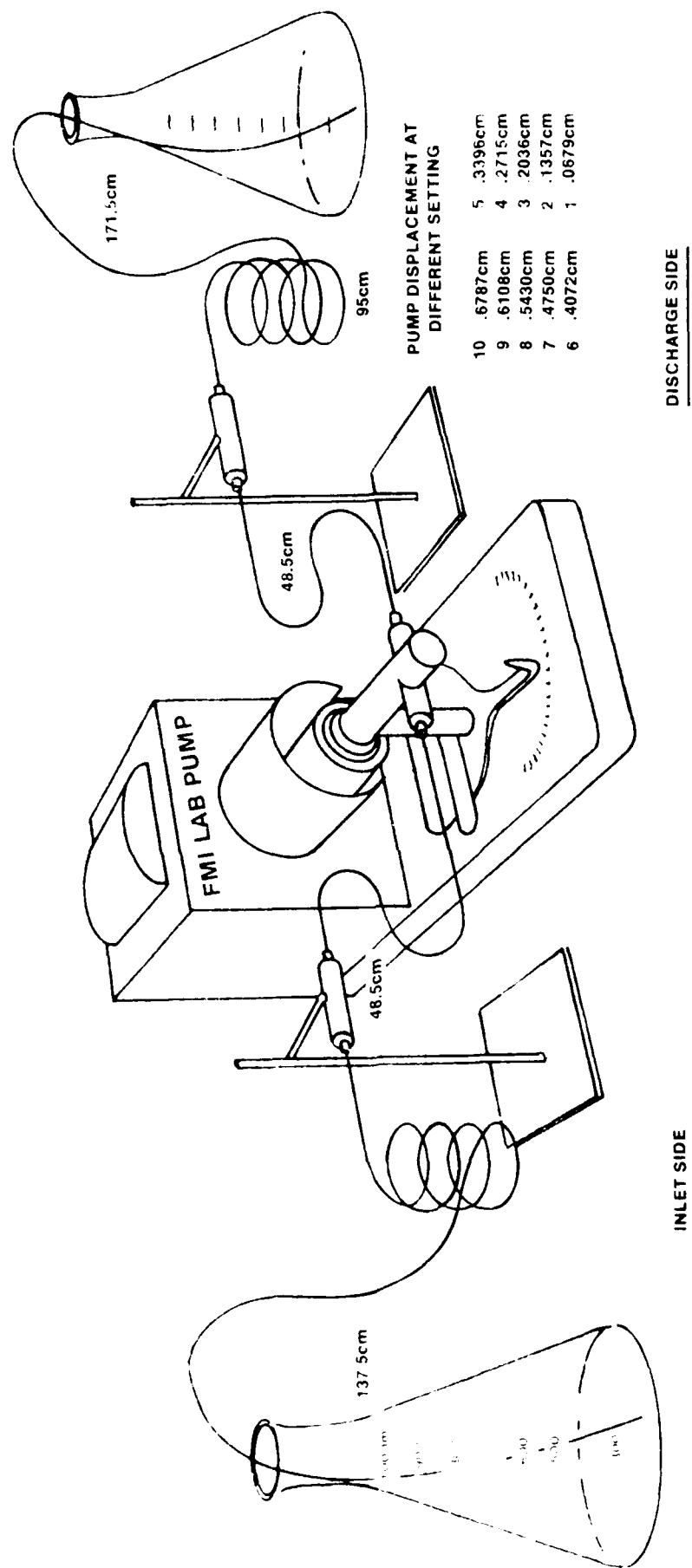
FIGURE 2.26 SCHEMATIC OF INERTIAL RHEOMETRIC PUMP TEST APPARATUS

60

... Pres... ... ...r of milliliters pumped per 188.4 seconds versus the pump
str... ... setti... ... ti... al plot is shown in figure 2.29.

... ... Results and Discussion.

The Inertial Rheometric Pump Test is able to measure the non-Newtonian flow
behavior of antimisting fuel and the shear thickening phenomena associated with
... ... The Inertial RP... ... provides a graphical display of the non-Newtonian
flow behavior of antimisting fuel. A typical antimisting fuel sample follows a
Newtonian flow pattern up to a stroke setting of 60 percent on the FMI lab pump.
However, above that ... percent stroke setting, there is a reduction in the volume
... ... with an increase in viscosity of the AMK, due to the fact that the
... ... shear rate of the AMK has been reached. Above the 70 percent stroke
... ... the AMK discharged from the pump appears to be in a gelled form, and the
... ... reverts to a fluid once it is in the discharge flask (reference 15).
The test ... has been shown to have the ability to distinguish between various
... concentrations of FM-9 polymer in AMK as shown in figure 2.30.

... there is no test to show the effect of using inline blended AMK versus
... blended AMK on the test results of the Inertial Rheometric Pump Test, and
... ... to assess any differences in test results caused by batch-to-
... variations in the FM-9 polymer which is commonly known to affect changes in
... ... rheory characterization tests. Due to the limited
... ... testing associated with this test method, it has not been routinely
... ... utilized as a ... test during the AMK program.

... ... AND VISCOSITY RATIO TEST.

... ... Background and Theory.

This test method covers the determination of kinematic viscosity and dynamic
viscosity of Jet A and AMK. This method is intended for application to Newtonian
... ... where the shear stress and shear rates are proportional.

... ... the viscosity is a measure of the resistive flow of a fluid under
gravity. For any viscometer, the time of flow of a fixed volume of fluid is
directly proportional to its kinematic viscosity, $v = \eta/\rho$, where η is the dynamic
viscosity coefficient and ρ is the density of the fluid. The kinematic viscosity
coefficient has the dimension L^2/T, where L is a length and T is a time, and is
frequently expressed in the units of centistokes, cSt.

... ... is the ratio between the applied shear stress and the rate
of shear. The dynamic viscosity, η, is thus a measure of the resistance to flow of a
fluid ... generally known as the viscosity of a liquid. The dimension of the
coefficient of dynamic viscosity is M/LT or FT/L^2, depending on whether the
... ... viscosity is based on the Mass (M) system or the Force (F) system.
... ... frequently expressed in centipoises, cP.

... ... a dimensionless number determined by the ratio of the
... ... time of AMK to pass through the viscometer
... ... the measured flow time of the AMK's base Jet A.

CC'S X 100
PUMPED IN 188.4 SECONDS

JET A = ●
AMK (0.3% FM9) = 0

PUMP SETTING
% OF CYLINDER STROKE

86-2?
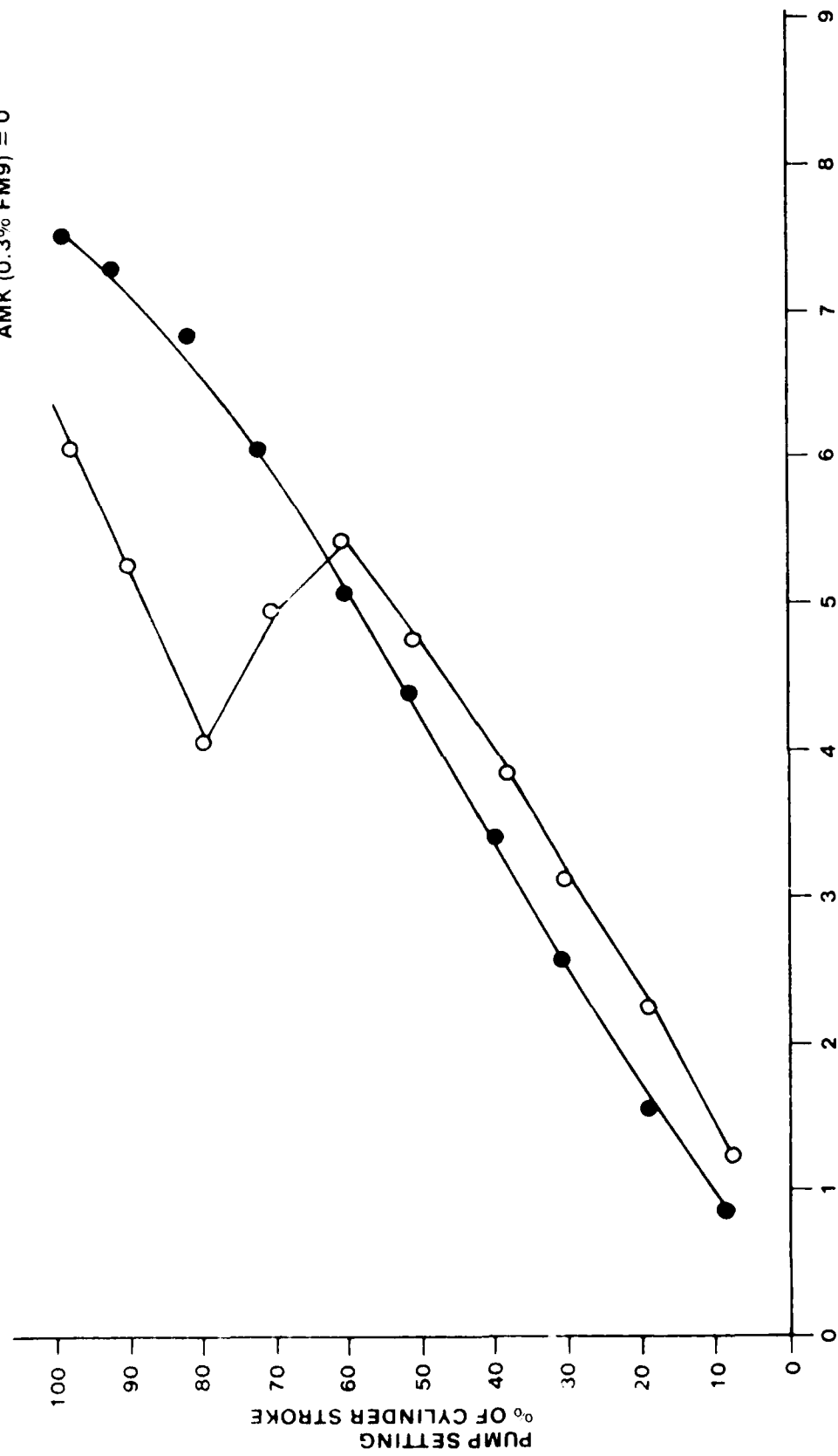
FIGURE 2.?? TEST DATA AND INERTIAL RHEOGRAM OF JET A AND AMK

FIGURE 2.30 INERTIAL RHEOGRAMS ON THE EFFECT OF FM-9 CONCENTRATION IN AMK

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2.11.2 Experimental.

2.11.2.1 Apparatus.

The apparatus used to determine the viscosity and viscosity ratio of AMK is a Cannon-Fenske Opaque Viscometer for transparent and opaque liquids and is shown in figure 2.31.

The Cannon-Fenske Opaque Viscometer is a reverse-flow viscometer that allows the liquid sample to flow into a timing bulb not previously wet by the sample. Using the viscometer, the time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of this calibrated viscometer, with a reproducible driving head pressure and closely controlled temperature (reference 16). The kinematic viscosity is the product of the measured flow time and the calibrated constant of the viscometer.

2.11.2.2 Equipment Specifications.

1. Calibrated Cannon-Fenske Opaque Viscometer, ASTM size number 75, viscosity range 1.6 to 8.0 centistokes (figure 2.31).

2. Viscometer holder to suspend viscometer in a vertical position.

3. Pipette, 15 ml.

4. Viscometer bath, deep enough so that the sample in the viscometer is at least 20 mm below the water surface and 20 mm above the bath bottom with a temperature controller. The temperature control must be such that for a range from 15 to 100° C, the temperature of the bath does not vary by more than 0.01° C over the length of the viscometers, or between the position of each viscometer and the location of the thermometer.

5. Timer, capable of reading tenths of a second.

6. Thermometer of suitable range.

7. Acetone.

2.11.2.3 Procedure.

1. Clean the viscometer by aspirating acetone through it, followed by vacuum drying.

    Note: Periodically, clean with chromic acid to remove traces of organic material.

2. Set the temperature of the viscometer bath to 25°C ±0.05°C.

3. Charge the sample into the viscometer as follows, using the diagram in figure 2.31 as a guide:

        a. Pipette 15 mls of sample into Tube N.
        b. Apply suction to Tube L, and draw liquid to the center of Bulb A.
        c. Stopper Tube N, and let set for 3 minutes.
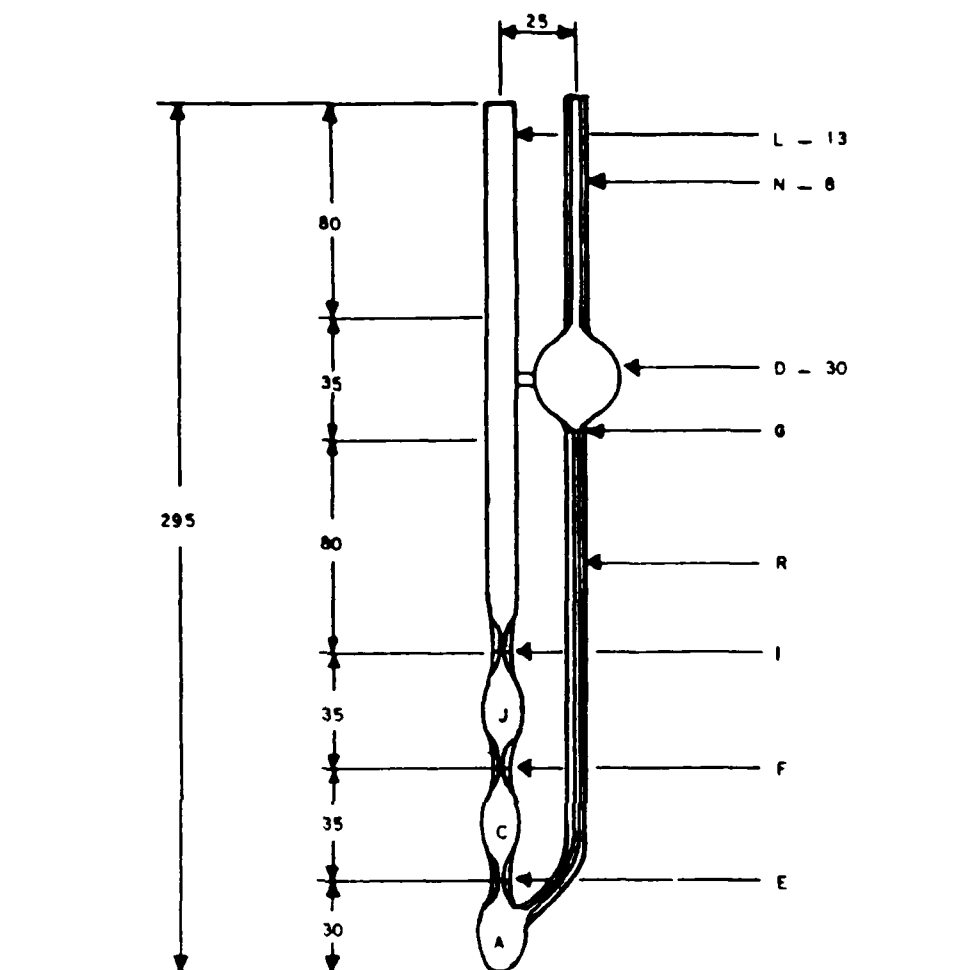
Size No	Approximate Constant, cSt/s	Kinematic Viscosity Range,* cSt	Inside Diameter of Tube R, mm (±2 percent)	Inside Diameter of Tube N Tubes E, F, and I, mm (±5 percent)	Volume, Bulbs A, C, and J, ml (±5 percent)	Volume, Bulb D, ml (±5 percent)
25	0.002	0.4 to 2	0.31	3.0	1.6	11
50	0.004	0.8 to 4	0.42	3.0	2.1	11
75	0.008	1.6 to 8	0.54	3.0	2.1	11
100	0.015	3 to 15	0.63	3.2	2.1	11
150	0.035	7 to 35	0.78	3.2	2.1	11
200	0.1	20 to 100	1.02	3.2	2.1	11
300	0.25	50 to 200	1.26	3.4	2.1	11
350	0.5	100 to 500	1.48	3.4	2.1	11
400	1.2	240 to 1 200	1.88	3.4	2.1	11
450	2.5	500 to 2 500	2.20	3.7	2.1	11
500	8	1 600 to 8 000	3.10	4.0	2.1	11
600	20	4 000 to 20 000	4.00	4.7	2.1	13

* 200 s minimum flow time for all units

NOTE — All dimensions are in millimetres

FIGURE 2.31 CANNON-FENSKE OPAQUE VISCOMETER FOR TRANSPORT AND OPAQUE LIQUIDS

4.   Align the viscometer vertically in the viscometer bath by means of a self-aligning holder.

5.   Allow 15 minutes for sample to come to temperature.

6.   Remove stopper from Tube N.

7.   Measure and record the flow times in seconds for the meniscus to pass from Mark E to Mark F.

8.   Draw the sample meniscus back into Bulb A and repeat steps 3, 4, 5, 6, and 7.

9.   Flow times should agree within 0.2 percent. If not, reject the test results and obtain additional flow times. If times are erratic, clean the viscometer, and run the sample again.

   NOTE: For viscosity ratio, run the neat jet fuel according to
   procedure steps 1-9, then repeat steps 1-9 with the sample.

10.   Determine the density of the sample to the nearest 0.001g/ml at 25°C using the procedure specified in section 2.16 of this report.

11.   Calculate the viscosity (kinematic then dynamic) and viscosity ratio and report to the nearest 0.01 CP and 0.1, respectively.

   a.   Viscosity

   Kinematic Viscosity (cSt)= Flow time (seconds) x viscometer
                                constant (see certificate of calibra-
                                tion that accompanies the viscometer for
                                the appropriate constant).

   Dynamic Viscosity (cP) =   Density (g/ml) x Kinematic viscosity.

   b.   Viscosity Ratio (Kinematic) (VR)

   $$VR = \frac{\text{Flow time of AMK FM-9 Sample}}{\text{Flow time of neat jet fuel}}$$

2.11.2.4   Results and Discussion.

The determination of viscosity depends on the behavior of the liquid sample and, ideally, the coefficient of viscosity should be independent of shear rate. This is commonly called Newtonian flow behavior. This is not the case for antimisting fuel which exhibits non-Newtonian flow behavior. The coefficient of viscosity for AMK varies with shear rate, different viscosities will be obtained from viscometers with different capillary diameters.

Shear viscosities have been measured in conventional gravity-flow capillary viscometers by ICI using the Cannon-Fenske Opaque Viscometer, by Southwest Research Institute using the Cannon-Ubbelohde viscometer, and by the University of California using the Oswald type viscometer. No evidence of antimisting fuel's non-Newtonian effects could be seen while the fuel was under the shearing conditions developed in these viscometers. For this reason, it was determined that these viscometers must be measuring the viscosity of AMK below the critical shear rate of AMK. Below the critical shear rate. the viscosity of AMK remains relatively constant and the AMK exhibits Newtonian flow behavior, but above the critical shear rate the viscosity of AMK varies and the shear thickening phenomenon of AMK becomes apparent. Since the non-Newtonian flow behavior (shear thickening effects) of AMK is of principal interest in the study of AMK, there has been little or no use of the conventional gravity-flow capillary viscometers. However, ICI has used the test method described above to measure the reproducibility of their batch products of a 0.30 percent FM-9 AMK in regards to viscosity and viscosity ratio. ICI has, in turn, set internal limits on the viscosity and viscosity ratio of 0.30 percent FM-9 AMK which are as follows:

Viscosity (dynamic)	Viscosity Ratio
2.45 to 2.65 cP	1.4 to 1.7

2.12 SOLIDS TEST METHOD.

2.12.1 Background and Theory.

The concentration of FM-9 polymer in Jet A fuel is of primary importance in the production of a quality AMK fuel. A quality AMK fuel is being defined as an antimisting fuel which exhibits the expected fire protection, degradability, and compatibility characteristics. In order to accurately determine the FM-9 polymer concentration of an AMK sample, the solids test has been used and the test has become one of the primary quality control tests for AMK.

The test method used for solids is standardized by American Society for Testing and Materials (ASTM) and is contained in ASTM designation D381-80, "Existent Gum in Fuels by Jet Evaporation." Existent gum, referred to as solid content in the case of AMK, is defined as the evaporation residue of an aircraft turbine fuel.

2.12.1.2 Apparatus.

A measured quantity of an AMK fuel sample is evaporated to remove the volatile material. This evaporation takes place with temperature controlled air or steam flow. The resulting residue, which is FM-9 polymer, is then weighed. The residue weight is compared to the original fuel sample on a weight-to-weight basis which results in the concentration of FM-9 polymer.

The standard ASTM test apparatus is schematically shown in figure 2.32. ASTM D381-80 specifies that the apparatus should be used with steam when conducting the test on aircraft turbine fuels. Even though the method standardized by ASTM works quite well and gives accurate results (reference 17), several other test
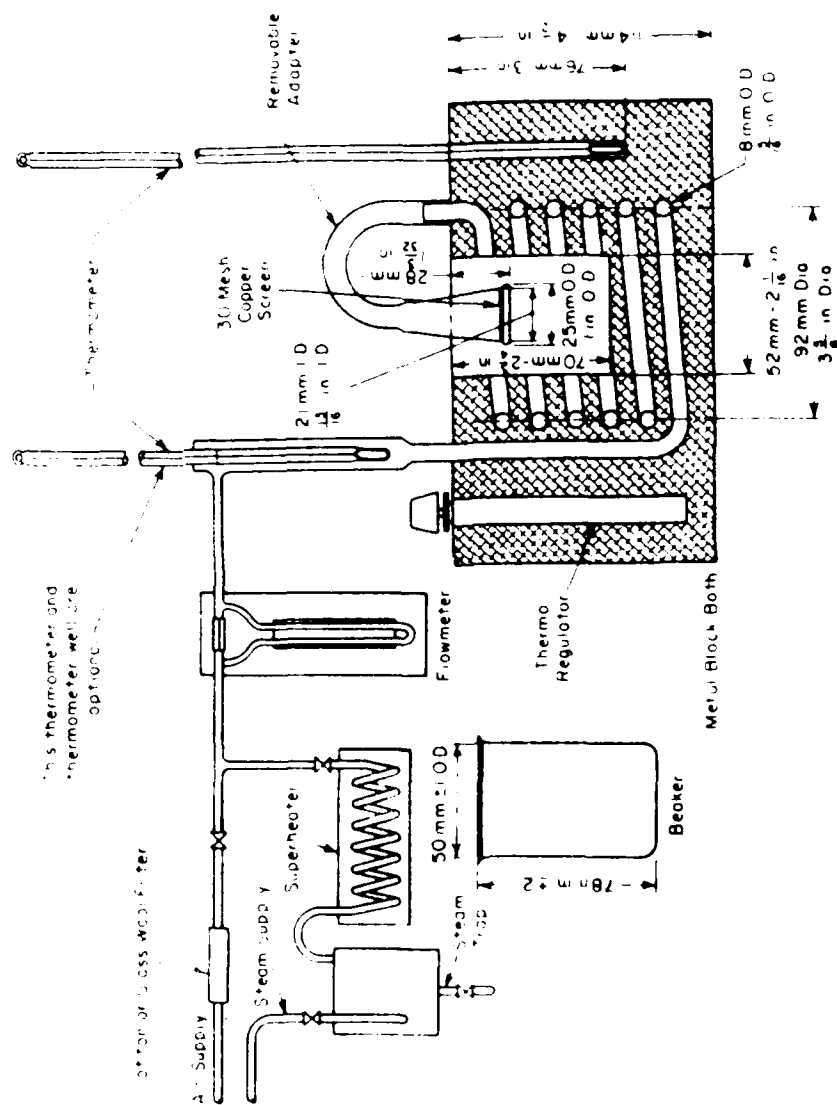
FIGURE 4.32 APPARATUS FOR DETERMINING EXISTENT GUM BY JET EVAPORATION

methods have been developed and used to determine the solids content of AMK. The FAA Technical Center uses a modified ASTM D381-80 test procedure, and the apparatus is shown in figure 2.33. The two major modifications of the test procedure consist of changing the vaporizing medium from steam to air and elevating the operating temperature of the bath. An entirely different method is used by ICI to evaporate the volatile materials. They use an apparatus known as an air friction oven. All of the above methods are based on the same principle. Only the means of evaporating the volatile materials are different.

2.12.2.2 Equipment Specifications.

2.12.2.2.1 ASTM Apparatus.

1. Balanced capable of reproducing weights to 0.1 mg.

2. Beakers, 100 ml capacity, dimensions shown in figure 2.32.

3. Cooling vessel, a desiccator without the drying agent.

4. Evaporation bath, either a solid metal block bath or a liquid bath, electrically heated and constructed in accordance with the general principals shown in figure 2.32. The bath should have wells and jets for two or more beakers. The rate of flow from each outlet when fitted with conical adapters should not differ from 1000 ml/s by more than 15 percent. A liquid bath, if used, shall be filled to within 25 mm (1 in.) of the top with a suitable liquid. Temperature may be maintained by means of thermostatic controls or by refluxing liquids of suitable composition.

5. Flowmeter, capable of metering a flow of air or steam equivalent to 1000 ml/s for each outlet.

6. Sintered Glass Filtering Funnel, coarse porosity, 150 ml capacity.

7. Steam Superheater, gas fired or electrically heated, capable of delivering to the batch inlet the required amount of steam at 232°C (450°F).

8. Thermometer, range -5 to +400°C.

9. Steam, supply of oil-free steam at a pressure not less than 5 psi.

10. Gum Solvent, 50/50 mixture of toluene and acetone.

2.12.2.2.2 FAA Modified ASTM Apparatus.

1. Five Unit Hi-Temperature Bronze Block Gum Bath, catalog number 74801, manufactured by Precision Scientific Company, Chicago, Illinois (see figure 2.33).

2. Beakers, 100 ml capacity, pyrex.

3. Desiccator without the drying agent.

4. Automatic Balance Meter, capable of reproducing weights to 0.1 mg.
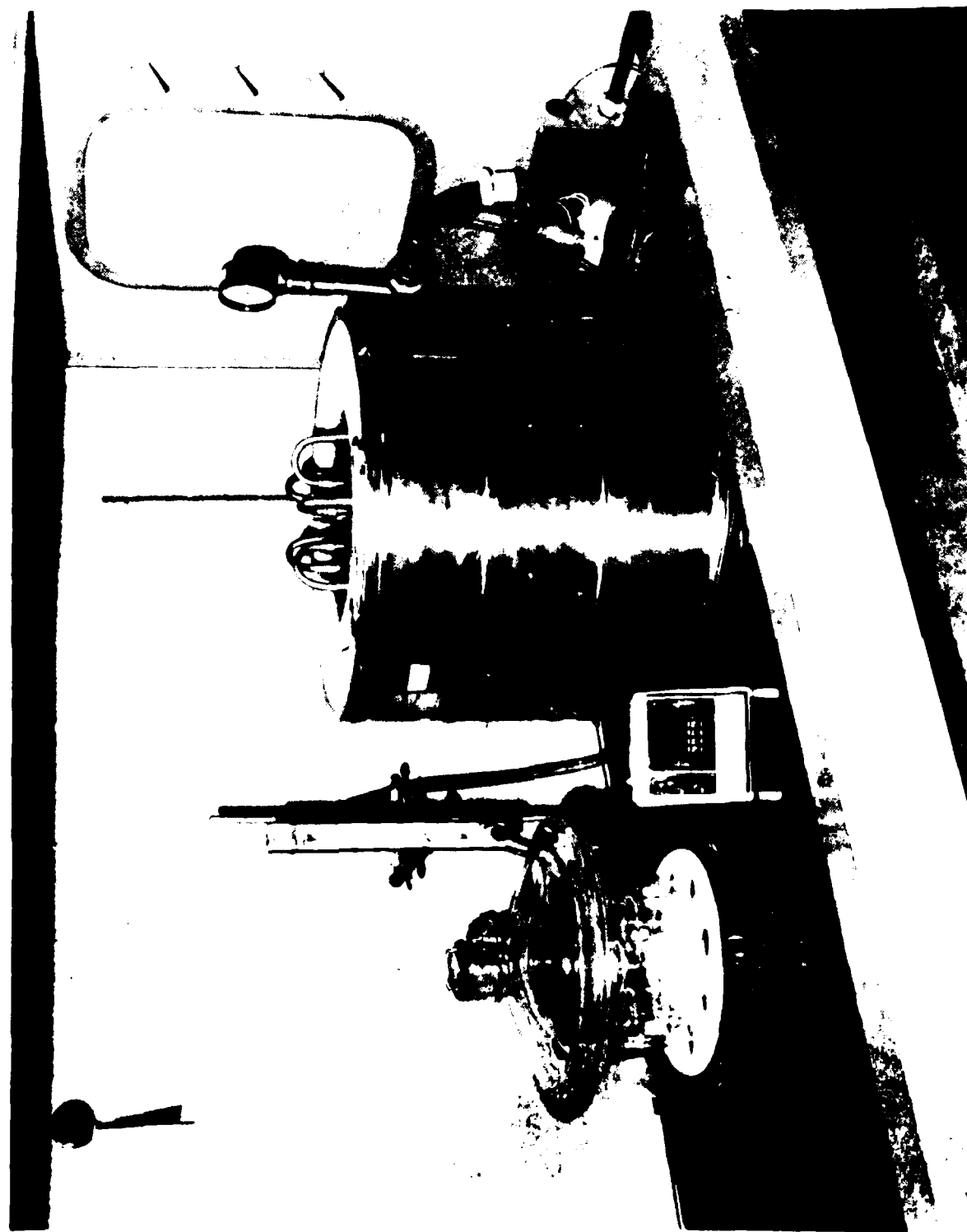
FIGURE 4.43 GAS SOLIDS TEST APPARATUS

5. Thermometer, range -5 to +400°C.

6. Sodium Hydroxide crystals.

7. Air supply which can be regulated to maintain 5 to 6 liters per minute airflow.

8. Air filter.

9. Flow Meter, Matheson Number 604.

10. Stainless steel forceps.

2.12.2.2.3 ICI Solids Test Apparatus.

1. Aluminum disposable weighing dishes, diameter 70 mm.

2. 30 ml beakers.

3. Air friction oven, Blue M Model POM-9-256-IFFG regulated at 150°C $\pm$2°C, damper temperature range adjustment at 2.

4. Oven, 110 $\pm$5°C.

5. Analytical balance.

6. Desiccator.

2.12.2.3 Procedures.

2.12.2.3.1 ASTM Procedure.

1. Assemble the steam-jet apparatus as shown in figure 2.32. The evaporation bath must be provided with an effective exhaust hood to control flammable vapors.

2. To place the apparatus in operation, apply heat to the bath. When the temperature reaches 232°C apply heat to the superheater and slowly introduce dry steam into the system until rate of 1000+150 ml/s for each outlet is reached. Regulate the temperature of the bath to a range from 232 to 246°C and the superheater to provide a well temperature of 232+3°C. Measure the temperature with the specified thermometer, placed with the bulb resting on the bottom of a beaker in one of the bath wells with the conical adapter in place. Any well having a temperature that differs by more than 3°C from 232°C is not suitable for standard tests.

3. Calibrate the flowmeter by successively condensing the steam flow from each outlet and weighing the water condensate. To accomplish this, attach a copper tube to a steam outlet and extend the tube into a 2-liter graduated cylinder that has been filled with crushed ice and then weighed. Exhaust the steam into the cylinder for approximately 60 seconds. Adjust the position of the cylinder so that the end of the copper tube is immersed in the water to a depth of less than 50 mm to prevent excessive back pressure. Weigh the cylinder. The gain in weight represents the amount of steam condensed. Calculate the steam rate as follows:

$$F = (W - w)1000/kt$$

where:

k = steam rate, ml/s of steam at 232°C,
W = weight of graduated cylinder with condensed steam, g,
w = weight of graduated cylinder and ice, g,
t = weight of 1000 ml of steam at 232°C at atmospheric pressure = 0.434g,
    and
t = condensing time, s.

      4. Adjust the flow to give a steam rate of 1000 ml/s for the outlet under test. Check the remaining outlets for uniform steam flow. Make necessary changes in individual outlets if the rate varies by more than 150 mL of steam/s. With all outlets adjusted to deliver 1000±150 ml of steam/s, record the flowmeter reading and use this setting for subsequent testing.

      5. Wash the beakers, including the tare, with the gum solvent until free of gum. Rinse thoroughly with water and immerse in detergent cleaning solution. Remove the beakers from the cleaning solution by means of stainless steel forceps and handle only with forceps thereafter. Wash the beakers thoroughly, first with tap water and then with distilled water, and dry in an oven at 150°C (302°F). Place the cooling vessel in the vicinity of the balance. Cool the beakers for at least 2 hours in the cooling vessel.

      6. Place the tare beaker on the right-hand pan of the balance and weigh the test beakers to the nearest 0.1 mg. When a single pan type balance is used, weigh the tare beaker as a blank. Record the weights.

      7. If suspended or settled solid matter is present, mix the contents of the sample's container thoroughly. At atmospheric pressure, immediately filter a quantity of the sample through a sintered-glass funnel of coarse porosity. Treat the filtrate as described in number 8. After determining the numerical gum value, designate by the word "filtered," the fact that extraneous material has been removed.

      8. By means of cylinders, add 5 ml of the sample to each beaker except the tare. Use a fresh beaker for each of the fuels to be tested. Place the filled beakers, including the tare, in the evaporation bath. The elapsed time between filling the first and last beaker in the bath must be as short as possible. Allow the system to warm for a minute before replacing the conical jet, which should have been brought to steam prior to attaching to the outlets. Center the jet to avoid disturbing the liquid. Maintain the temperature and rate of flow and allow the sample to evaporate for 30 minutes. Samples tested together should have similar evaporation characteristics.

      9. When the evaporation is complete, transfer the beakers from the bath to the cooling vessel and cool for at least 2 hours. Weigh the beakers in the same manner as before and record the weights.

      10. Follow the procedure of ASTM D 381 to calculate the existent gum content of the test fuel in milligrams per 100 milliliters. To determine the concentration of film polymer in JFK, the

standard calculations are changed to yield a concentration on a weight-to-weight basis. The calculation of FM-9 polymer concentration in AMK is as follows:

$$\frac{(\text{weight of beaker and residue} - \text{weight of beaker})}{(\text{weight of beaker and sample} - \text{weight of beaker})} \times 100 =$$

% polymer or % concentration

2.12.2.3.2   FAA Modified ASTM Procedure.

1.   Assemble the air-jet apparatus as shown in figure 2.33.   The evaporation bath must be provided with a fume hood to remove flammable vapors.

2.   Turn on the switch marked "INT" of the Hi-Temperature Bronze Block gum bath.   This switch supplies power to the controller pyrometer which, in turn, controls the relay and the 3-ring heaters.

3.   Set the temperature controller to regulate the temperature of the bath to a range from 232° to 246°C.

4.   Turn on the air supply which must be filtered.   Regulate the airflow into the bath to 5 to 6 liters per minute with all air nozzles in place.

5.   Measure the temperature of each test well.   This should be done with the bulb of the thermometer resting on the bottom of a sample beaker placed in one of the test wells with the air nozzle in place.   The temperature should be 232±3°C.   Any well having a temperature that differs by more than 3°C from 232°C is not suitable for use in conducting this test.

6.   Clean the beakers by placing half a teaspoon of sodium hydroxide crystals in the beaker and filling it with water.   Allow this mixture to stand overnight, then rinse the beaker with tap water and wash with detergent and rinse again.

7.   Condition the beakers in an oven at 150°C for approximately 30 minutes and then allow them to cool to room temperature in the desiccator.   NOTE: At this point, the beakers should never be handled.

8.   This procedure calls for four beakers to be used each time the test is done.   Number each beaker and weigh them on the automatic balance to three decimal places.   Record the beaker number and weight.

9.   Add 40 ml of sample fuel to each beaker.   Beaker 1 and 2 should contain the AMK test sample and beaker 3 and 4 should contain known standard percent AMK. The known standard percent AMK is used to verify the accuracy of the test results each time the test is done.

10.   Again weigh each beaker and record the weight.

11.   Remove the air nozzles and place the filled beakers in the evaporation bath and reinstall the air nozzles.   Allow the sample to remain in the bath for period of one hour.

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After the evaporation period, remove the beakers using stainless steel
_____ in the desiccator for a period of one hour. The
_____ without the drying agent.

After the cooling period, reweigh the beakers and record the weight.

Calculations:

$$\frac{(\text{weight of beaker and residue} - \text{weight of beaker})}{(\text{weight of beaker and fuel} - \text{weight of beaker})} \times 100 =$$

% polymer or % concentration

_____ result for beaker 3 and 4 should be compared to the known
_____ in the AMF sample. If the percent concentrations are the same,
_____ needs to be done. However, if the experimental result is higher
_____ the known concentration, the AMF test sample concentration should
_____ accordingly.

_____ Solids Test Procedure.

_____ aluminum dishes in a 105°C oven for 30 minutes and
_____ temperature in desiccator.

_____ nearest 0.0001 gm).

_____ imately 10 grams of sample from a 30 ml beaker to the
_____ the weight.

_____ an air friction oven at 150°C for 2 hours. If
_____ control, shift the samples to eight, and place in the oven on
_____ for circulation.

_____ place in desiccator for 5 minutes, then weigh.

_____

$$\frac{\text{_____} - \text{weight of dish}}{\text{_____} - \text{weight of dish}} \times 100 = \% \text{ Total Solids}$$

_____

_____

_____ using several different test methods, all of which
_____ procedures are followed exactly. All of the test
_____ the volatile materials of an AMF sample and
_____ Duplicate tests should always be conducted on
_____ these test methods is ±0.01 percent
_____ are conducted. Some typical solids test
_____ AMF standard methods are shown in table 2.2.

TABLE 2.2 STEAM JET GUM* ANALYSIS OF AMK SAMPLES
(FUEL SPILLAGE TESTS)

FAA Technical Center Test Number	Wt% FM-9	
	Nominal	Gum**
1	0.40	0.358
2	0.40	0.351
3	0.40	0.360
4	0.40	0.357
5	0.40	0.355
6	0.40	0.360
9	0.40	0.397
10	0.267	0.270
11	0.267	0.262
12	0.267	0.264
13	0.333	0.328
14	0.333	0.331
15A	0.333	0.346
22	0.30	0.303
23	0.30	0.304
24	0.30	0.292
25	0.30	0.306
26	0.30	0.304
27	0.30	0.300
30	0.30	0.301
31	0.30	0.295
33	0.20	0.195
34	0.20	0.194
36	0.20	0.194
38	0.20	0.193
41-44	0.30	0.296
45	0.30	0.302
49-53	0.250	0.228
55	0.30	0.287
59-61	0.26	0.265
62-64	0.26	0.267

* ASTM D-381
** Average of Duplicate Samples

These test procedures can also be used to determine the polymer content of FM-9 slurry. To accomplish this, the AMK test sample is replaced by FM-9 slurry. The amount of FM-9 slurry used is the same as the AMK test sample; and the same test procedures are followed.

The solids test is one of the primary quality control tests for inline blended AMK because it can accurately measure the FM-9 polymer concentration of AMK. The FM-9 polymer concentration is important because it can affect the fire protection, degradability, and compatibility of antimisting fuel.

... use of FN-9 antimisting fuel is the tendency of the fuel ... gels, and precipitates when in contact with water in either ... The amount of water dissolved in Jet A fuel at any given ... ... by equilibration with the water in the atmosphere ... (reference 18). The water solubility of Jet A fuel is 52 parts ... the saturation temperature of 22°C (reference 18). ... its water saturation point, the fuel tends to shed the ... form, depending on the temperature. As this process ... interface forms between the fuel and the water which ... This water can then be removed by draining fuel tank ... through a coalescing fuel filter (water separator).

... affinity for water and the FM-9 polymer can act as an ... (18). The amount of dissolved water that antimisting ... reater than the amount Jet A can hold at any given ... whereas FM-9 AMK can hold up to 1300 ppm of water at 22°C ... can only hold up to 52 ppm at 22°C. Experiments have ... increase in the water content of AMK when in ... or vapor form and the normal saturation-drying ... does not occur for AMK. The uptake of water by ... period of time is shown in figure 2.34. Even though ... large quantity of dissolved water, once AMK reaches ... point, it tends to become cloudy and the polymer tends to ... this, there was concern for the potential of clogging ... gel within an aircraft fuel system. In order to ... potential problem area, it became necessary to make water ... and AMK samples.

... termination of water content in Jet A and ... Fischer Titrations. Concentrations of water from 10 to ... be determined when a sample of Jet A or AMK is ... Fischer reagent to an electrometric end point. This ... constant direct current (DC) voltage (biamperometry ... current (bivoltametry) to a double ... in the titration mixture. During the titration, the ... observed. The basic ingredients of the Karl Fischer ... iodide and the background solvents may be ... methanol, pyridine, etc. In the presence of water, ... the iodine to sulfuric acid in accorandce with ... equation:

$$\ldots \longrightarrow 2\mathrm{HI} \cdot 2\mathrm{HI} + \mathrm{RN} \cdot \mathrm{HSO_4CH_3}$$

... monitored by the background solution when no more ... mixture, the excess of free iodine acts as an ... in a sharp voltage or current jump, thus ... end point.

FIGURE 2.34   UPTAKE OF WATER BY FM-9 AMK

2.13.2   Experimental.

2.13.2.1   Apparatus.

The apparatus for determination of water by using Karl Fischer reagent is shown in figure 2.35 and contains a microammeter, titration flask with integral magnetic stirrer, reagent reservoir, buret, electrodes, battery, and potentiometer.   This method measures current flow in a solution between platinum electrodes connected in circuit with a microammeter and a DC voltage source.   The electrodes are placed in the sample solution, a polarizing voltage is applied, and the Karl Fischer reagents slowly added by use of the buret.   In the continued presence of water and spent titrate, all current carrying iodine will have been reduced to iodine and, thus, there will be no indication on the microammeter.   As more titrate is added to the sample, the end point is eventually reached when no more water is left in the sample to react with the iodine.   Any additional titrate simply adds highly conductive free iodine, which causes the microammeter to register a sharp increase in current flow which indicates the end point.

The titrate volume used to reach the end point is then used to calculate water content in the sample.

Drying Tubes

Reservo

Buret

Microammeter

Electrodes

Potentiometer

Switch

Battery

Magnetic Stirrer

DETERMINATION OF WATER BY KARL FISCHER REAGENT

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Since atmospneric moisture will cause erroneous results in Karl Fischer
titration, the system should be kept sealed and as dry as possible. Drying tubes
filled with indicating drier should be used in all air inlet lines.

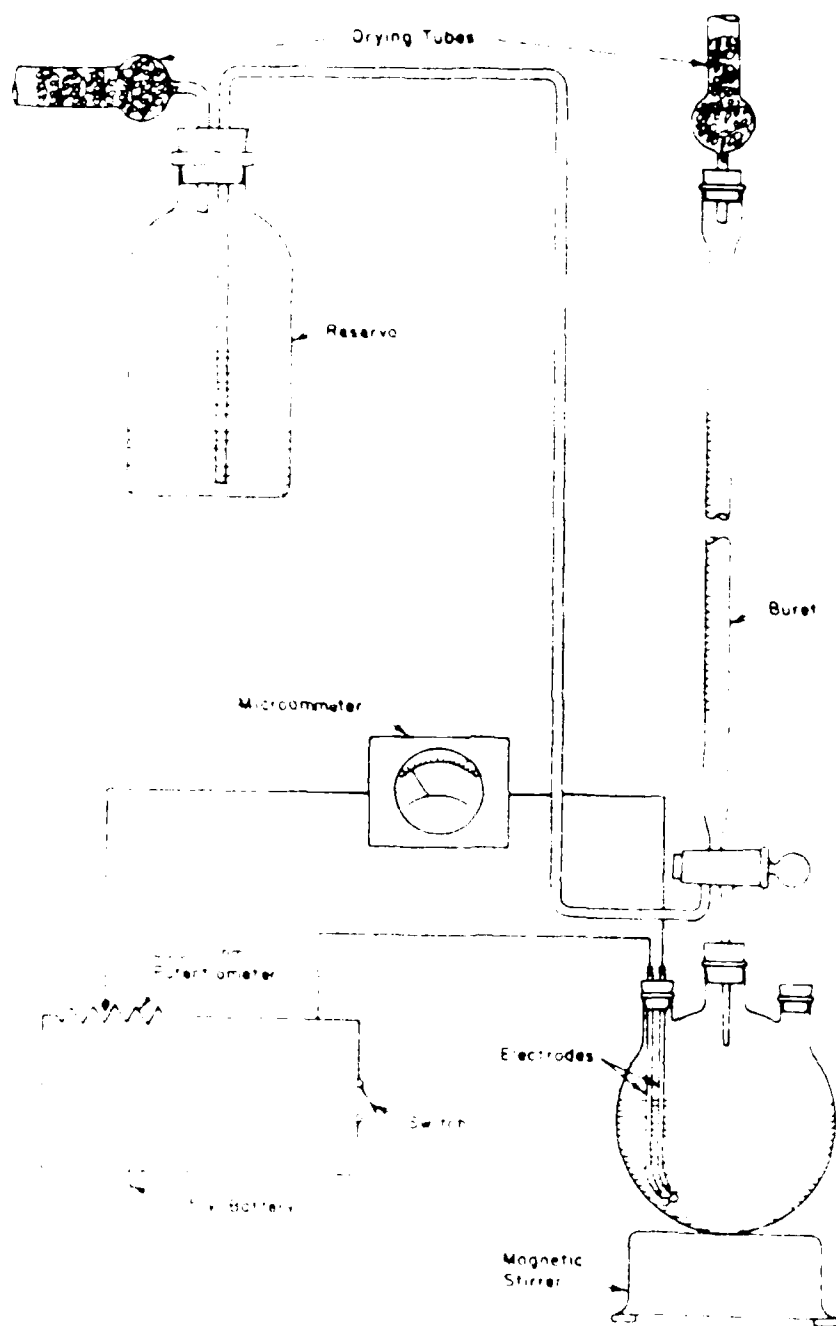### 2.13.2.1 Equipment Specifications.

Any apparatus designed for the purpose of conducting a titration with Karl
Fischer reagent can be used to determine the water content of an antimisting fuel
or Jet A sample. During the course of the AMK program, the FAA and its'
contractors used several different models of Karl Fischer titrators. The
titrators used during the program, most of which are automatic, are shown below.

ORGANIZATION	TITRATOR MODLE	MANUFACTURER
FAA	1. General apparatus specified by ASTM	
	2. 391 K-F Titrimeter	Fischer
	3. Metrohm 658	Brinkman
JPL	K-F Titrimeter-Automatic System	Fischer
PWA	702 Aquatest iI	Photovolt Corp.
Boeing	Aquatest IV	Photovolt Corp.
ICI	Auto-Aquatrator 68830	Precision Scientitic Co.

(See Note 1)

NOTE: The Auto-Aquatrator was modified by ICI to better adapt the equipment to
their needs. The following modifications were made:

1. The drying tubes and pumping systems were removed and replaced with a
completely enclosed system, using N_2 pressure.

2. The 15 ml buret was changed to an automatic zero 25 ml buret. The
original buret was filled at the top with the excess reagent removed by nitrogen
pressure at the base and the overflow drains into a reservoir. The buret is
calibrated in 0.05 ml increments.

3. The original beaker and hard rubber top was replaced with an improved,
CEL designed, all glass, one-piece vessel. The top of this vessel contains
ground-glass joints to accommodate the various components (electrode,
thermometer, buret tip, and sample port. The reservoir has a drain tube with
stopcock for easy removal of spent reagent and sample.

4. A small combination hot plate-magnetic stirrer replaces the magnetic
stirrer supplied with the unit. Similar to Thermolyne Model SP-10105B.

The titration assembly specified by ASTM in standard D1744-64, "Water in Liquid
Petroleum Products by Karl Fischer Reagent," is shown in figure 2.35 and consists
of the following equipment.

1. 10 ml Buret, graduated in 0.05 ml subdivisions and fitted within a 3-
way flash stopcock.

2. Reagent reservoir, any convenient size.

FIGURE 36 TITRATION ASSEMBLY ELECTRODE
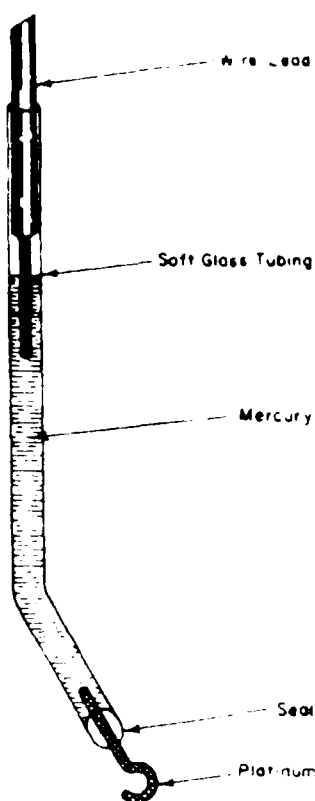
3. Magnetic stirrer.

4. Titration flask, a 3-neck flask of approximately 500 ml capacity.

5. Battery, 1.5 volt dry cell.

6. Potentiometer, resistance of 2000 ohm.

7. Micrometer.

8. Drying tubes, 2 required.

9. Indicating desiccant.

10. Electrodes, 2 required, typical electrode is shown in figure 2.36.

11. Miscellaneous wire, tubing, glass tubing, stoppers, etc.

12. Reagents

a. Karl Fischer Reagent, commercially prepared reagent may be used or
the reagent may be prepared by the following procedure: For each liter of
reagent, dissolve 85+1 g of iodine in 270+2 ml of pyridine in a dry, glass-
stoppered bottle. Add 670+2 ml of methanol (99.9 percent). Cool the mixture in
an ice bath to below 3.9° C. Bubble gaseous sulfur dioxide (SO_2) through
concentrated sulfuric acid (H_2SO_4, sp gr 1.84) into the cooled mixture. Continue
the addition of SO_2 until the volume is increased 50+1 ml. Alternatively, add
50+1 ml of freshly drawn liquid SO_2 in small increments to the precooled mixture
in an ice bath. Mix well and set aside for at least 12 hours before using.

b. Dilute Karl Fischer Reagent, adjust the strength of the full
strength reagent to a water equivalence of 2 to 3 mg of water per ml by dilution
with pyridine.

c. Sample solvent, mix 1 volume of methanol with 3 volumes of
chloroform.

2.13.2.3 Procedure.

The procedure for the various automatic Karl Fischer titrators varies from unit
to unit, for this reason, only the procedure specified by ASTM in D1744-64 for
the general Karl Fischer titration assembly will be given in this section.

1. Standardization of Karl Fischer Reagent.

(a) The dilute Karl Fischer reagent should be standardized at least
daily in accordance with 1.1.1 to 1.1.3.

(1) Add 50 ml of the sample solvent to a clean, dry titration
flask. Insert the stoper and adjust the magnetic stirrer to give a smooth stir-
ring action. Turn on the indicating circuit and adjust the potentiometer to give
a reference point with approximately 1 A of current flowing. Add Karl Fischer
reagent in suitable amounts to the solvent to cause the needle to deflect from
the reference point. At first, the needle will deflect due to local
concentration of the unreacted reagent about the electrodes, but will fall back
to near the reference point. As the end-point is approached, the needle will
fall back more slowly after each addition of Karl Fischer reagent. The end-point
is reached when, after the addition of a single drop of reagent, the needle
remains deflected at least 1 A from thne reference point for at least 30 s.

(2) To the solution in the titration flask, carefully add from a
weighing pipette previously weighed to the nearest 0.1 mg, 1 drop of distilled
water. Stopper the flask. Reweigh the weighing pipette. Titrate to the end-
point as described in (1).

(3) Calculate the water equivalence of the Karl Fischer reagent as
follows:

$$F = W/T$$

where:

  F = water equivalence of Karl Fischer reagent, mg/ml,
  W = milligrams of water added, and
  T = milliliters of reagent required for titration of the added water.

2. Sample Titration

   a. Add 50 ml of solvent to the titration flask and titrate with standardized Karl Fischer reagent to the end-point as described in 1.1.1. It is important to stopper the sample inlet tube as qickly as possible to prevent absorption of moisture from the atmosphere.

   b. Obtain an hydrometer reading on the material to be tested and convert degrees API to specific gravity without correcting for temperature. Immediately pipette 50 ml of the sample into the titration flask. Alternatively, the sample may be weighed and added to the titration flask in any convenient manner.

   c. Titrate the sample to the end point as described in (1). Record the milliliters of reagent used.

3. Calculation.

   a. Calculate the water content of the sample as follows:

$$WATER, PPM = (CF \times 1000)/W$$

where:

  C = milliliters of reagent required for titration of the sample,
  F = water equivalence, milligrams mg water/ml,
  1000 = factor for converting to parts per million,
  W = grams of sample used = A X G,
  A = milliliters of sample used, and
  G = specific gravity of the sample.

2.13.2.4 Results and Discussion.

All of the Karl Fischer titrators listed in the equipment specifications section of this test method produced acceptable water content measurements of Jet A and AMK samples after an initial problem of AMK gellation on the electrodes of the titrators was corrected. This gellation problem plagued all of the titrators when water content measurements first began to be made on AMK samples. The gel that formed on the electrodes of the Karl Fischer titrators caused water content measurements that were erratic and much higher than would normally be expected.

Researchers found several possible solutions to this problem. Pratt and Whitney Aircraft (PWA) thought that the gel formation was caused by the magnetic stirring of the virgin AMK sample. The magnetic stirrer was thought to produce enough shear stress to cause the virgin AMK sample to gel. They determined that this problem could be alleviated if the virgin AMK sample was degraded before the sample was titrated in the Karl Fischer titrator (reference 19).

The Jet Propulsion Laboratory (JPL) had a different theory about the gel formation on the electrodes. Their theory was that the gel was caused by the presence of methanol in the commercially prepared Karl Fischer reagent. The reaction between the methanol and the FM-9 polymer causes the FM-9 polymer in the antimisting fuel sample to precipitate and coat the electrodes of the titrator with gel. This gel causes the erratic water content measurements for AMK samples. JPL determined the problem could be avoided by adding 50 cc of pyridine to the AMK sample prior to titration (reference 20). The pyridine is added in order to keep the FM-9 polymer in solution while it is in the presence of methanol.

Boeing Military Aircraft Company also tried several approaches in an attempt to find a solution to this problem of gel formation or the electrodes of the Karl Fischer titrator. The first approach was to add a solvent to the titration vessel to increase the solution volume. Chloroform was used as the solvent. The other approach was to use a larger volume of titration solution in the titration vessel and to change the solution more frequently than recommended in the procedure for their titrator (reference 17).

The modified procedure for Karl Fischer titrations, which was finally used to measure the water content of AMK samples accurately with repeatability and confidence, consisted of making the following changes in the operational procedure of the titrators:

1. To reduce the concentration of AMK in the titration vessel, the volume of solvent placed in the titration vessel was increased.

2. To reduce the build up of FM=9 polymer in the titration vessel caused by residual AMK samples, the solvent in the titration vessel was changed frequently.

2.14 GLYCOL MEASUREMENT.

2.14.1 Background and Theory.

FM-9 polymer in a virgin state is a dry, granular powder. In order to give the FM-9 Polymer flow characteristics so that the inline blending process could be developed, ICI combined the polymer with a carrier fluid to form what is known as FM-9 slurry. The carrier fluid consists of glycol and amine. Standard "Avgard " FM-9 polymer based slurry consists of the following constituents:

CONSTITUENT	PERCENT OF TOTAL SLURRY WEIGHT.
FM-9 Polymer	25 + 0.5
Amine	1.25 + 0.25
Water	1.25 + .25
Glycol	Remainder

Researchers felt that the glycol/amine carrier fluid increased the effectiveness of the FM-9 polymer relative to the mist suppression and resulting fire protection capabilities of antimisting fuel (reference 21). It became apparent that there was a need to verify by analysis the amount of glycol and amine present in the antimisting fuel samples in order to evaluate the effects of the carrier fluid.

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spectroscopy to measure the amount of glycol in anti-
... also developed a method for ...
... in the AMK using a procedure that combined extraction
... infrared spectroscopy.

... of the infrared spectrophotometer was conducted using glycol
... concentrations that were specially prepared in a xylene,
... to avoid any possible interference of the PY-0 polymer or the
... absorbance readings. The optimum wavelength for measuring the
... was determined to be 3542 cm^{-1}. The absorbance at that
... to correlate with the weight percent of glycol in a sample.
... Figure 2.37 shows there is a linear correlation between the weight
... sample and the absorbance of the sample. Further testing
... determined that the absorbance factor was not affected by
... using the curve shown in figure 2. ... would be used
... weight percent of glycol present in an AMK sample.

... Equipment.

... Operation.

... Infrared Spectrophotometer combines double-beam
... microprocessor hardware and associated software
... data processing.

... are available in two modes, Scan and Quantitative. Spectra may
... pinpointed using single-step and peak-pick routines.
... may be made at selected wavelengths using a point
... AMK analysis, a single-step point program routine was used.

... with the AMK test sample is placed in the sample chamber of
... spectrometer and the single-step point program routine automatically
... transmittance at the chosen wavelength for a specified number of
... averages the readings and gives a percent transmittance (%T)
... value can then be calculated and used to determine the
... of the AMK sample from the curve shown in figure 2.37.

... equipment specifications.

... computing spectrophotometer.

... liquid sample cell.

... specific surface.

... Reagents.

... reagent grade.

... reagent grade.

... distilled water.
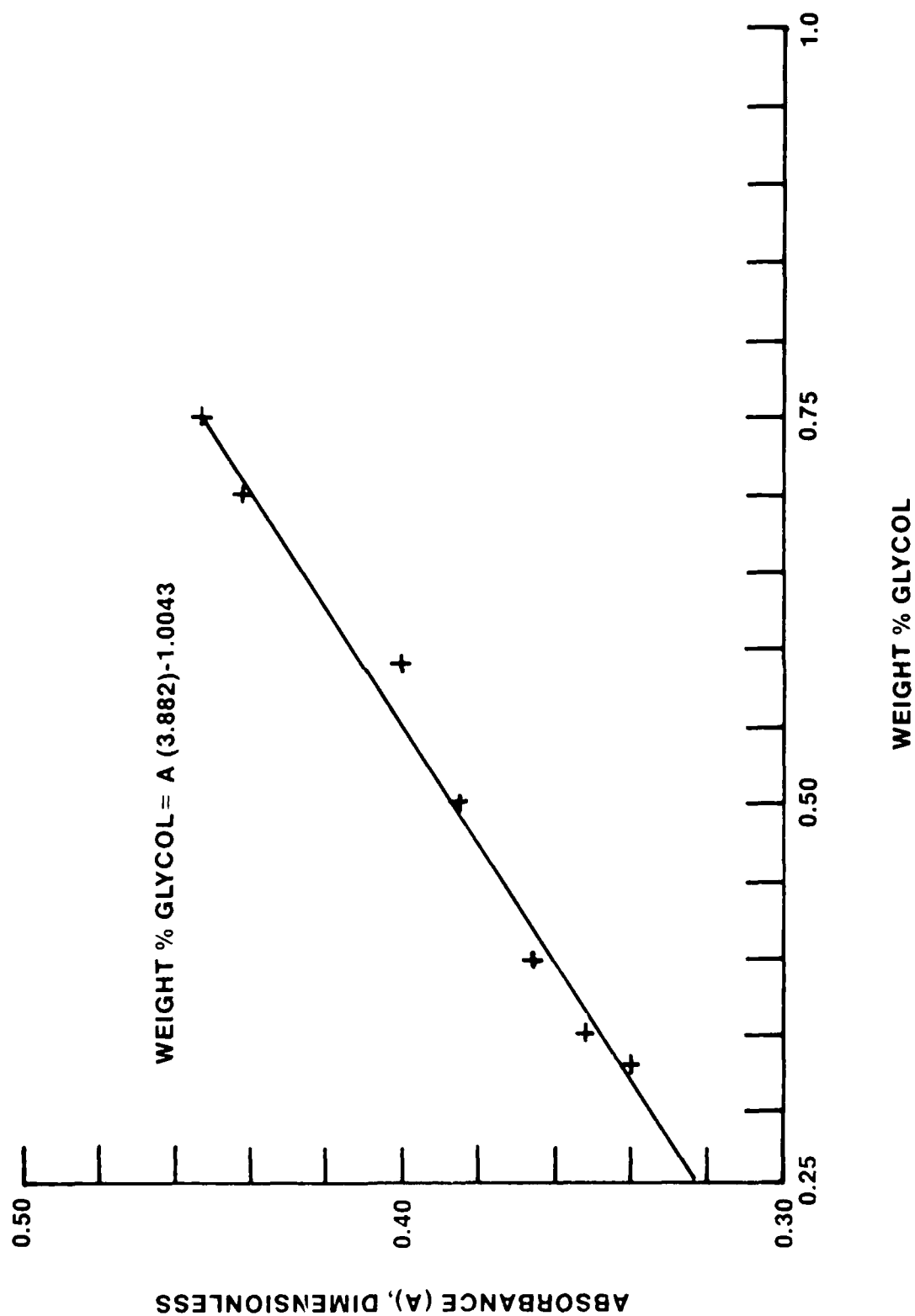
FIGURE 2.37 WEIGHT PERCENT GLYCOL VS ABSORBANCE FOR GLYCOL ABSORBANCE

_____ Procedure.

_____ specify _____ instructions and operation _____ in the Beckman
_____ of the manual).

_____ select the single-step point program routine for the operation mode of
most interest.

_____ the following specifications into the program:

_____ = _____ cm⁻¹

_____ of readings = 10

_____ hypodermic syringes, fill the liquid sample cell with the
_____

_____ the sample cell into the sample chamber of the spectrophotometer.

_____ the spectrophotomer routine. NOTE: Values of percent trans-
_____ _____ approximately 2 percent over 10 minutes in the sample cell, so
_____ _____ is necessary.

_____ the percent transmittance value from the spectrophotometer.

_____ the absorbance of the AMK test sample as follows:

$$\text{Absorbance} = \log 1/\text{Transmittance}$$

_____ the weight percent of glycol in the AMK test sample using the
_____ weight percent glycol in AMK versus absorbance of AMK (figure 2.37).

_____ _____ Results and Discussion.

_____ content of antimisting fuel can be readily determined by infrared
_____ _____. There is a linear correlation between the weight percent glycol in
_____ sample and the absorbance of the AMK sample. SWRI determined the repeat-
_____ _____ this test method by conducting 10 runs of the same AMK test sample.
_____ results have a 2 percent standard deviation relative to the mean glycol
_____ _____ the AMK test sample (reference 2).

_____ _____ different technique to determine the glycol content of antimisting
_____ _____ Fourier Transform Infrared Spectrom _____. They used the technique of
_____ subtraction based on a calibration curve (reference 10). The
calibration curve was determined using samples of known glycol content prepared
_____ the same glycol used in FM-9 AMK. JPL also used this procedure to determine
the _____ content of an AMK sample. By subtracting the spectrum obtained for
virgin AMK from the spectrum for AMK that had water added, the difference in
spectra was found to correspond to the amount of water added to the AMK. A
calibration curve was constructed based on the difference in spectra from AMK
samples of known water content. The curve was then used to determine the water
content of AMK test samples.

2.15. AMINE MEASUREMENT.

2.15.1 Background and Theory.

For general background information on the reason a test method was developed to measure the amine content of antimisting fuel, refer to the Sulfur Measurement Section (2.14) of this report. A test method for determining the amount of amine present in an antimisting fuel sample was developed by SWRI based on the fact that when amine undergoes combustion, one of the products of combustion is nitrogen. The test method combines combustion of the AMK sample in a Pyrolysis furnace with nitrogen detection in the products of combustion by chemiluminescence.

The initial testing of this test method was conducted using standards that were prepared by diluting a stock solution of dimethylformamide (DMF) in toluene. The nitrogen content of the standards were determined by the following calculation (reference 2):

   1. Molecular Weight DMF (C_3H_7NO) = 73.11 g

   2. Wt% nitrogen in pure DMF = (14.0/73.11) x 100 = 19.16

   3. Wt% nitrogen in 0.1017 wt% stock solution = 0.001017 x 19.16 = 0.01949 (194.9 ppm)

   4. PPM (Nitrogen) in diluted standards:

         2 ml of stock solution diluted to 100 ml = 3.9 ppm
         4 ml of stock solution diluted to 100 ml = 7.8 ppm
         6 ml of stock solution diluted to 100 ml = 11.7 ppm

The chemiluminescence detector measures the nitrogen content in number of counts displayed on a digital readout. The counts for nine consecutive injections of the three standards shown in the above calculation were used to establish a calibration curve for number of counts versus the nitrogen content of an AMK sample. The calibration curve is shown in figure 2.38.

2.15.2 Experimental.

2.15.2.1 Apparatus.

AMK samples are prepared by diluting them with tetrahydrofuran (THF). The AMK samples are diluted to reduce plugging them in microsyringe which is used to inject the sample into the furnace. The plugging is caused by the evaporation of the Jet A and the formation of solid polymer in the needle tip of the microsyringe due to the 950° Celsius temperature of the furnace (reference 2). The diluted AMK samples are injected, using the microsyringe, into the pyrolysis furnace where the nitrogen compounds are oxidized and then measured as NO_2 by the chemiluminescence detector. The digitally displayed counts for AMK are compared to a standard nitrogen calibration curve to determine the nitrogen content. From the nitrogen content of the sample, the amount of amine present in the AMK sample can be calculated.
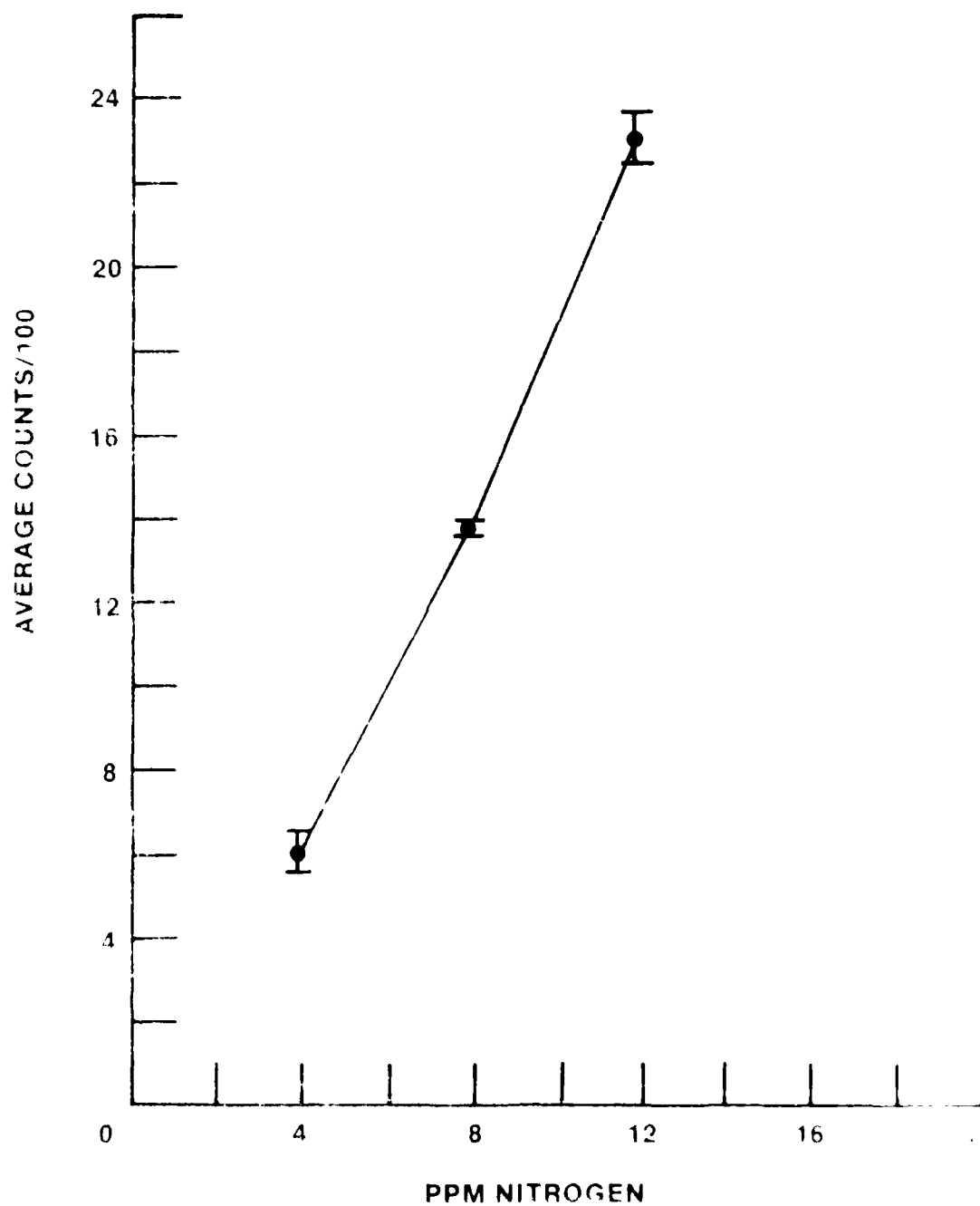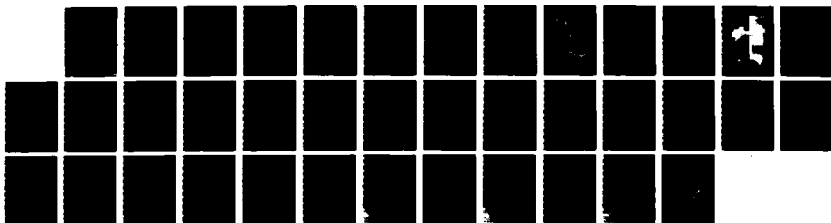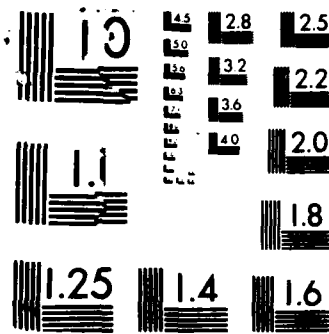
2.15.2.2 Equipment Specifications.

1. Antek Model 771 Pyro-reactor.

2. Antek Model 720 Nitrogen detector.

3. Microsyringe, 50 1 with 2" X 26 gage needle.

4. Constant rate syringe drive.

5. Silicone Septa.

6. Pipettes, Class A, 1 ml, 4 ml.

7. Quartz combustion tube, as per instrument manufacturer's design.

8. Reagents:

   8.1 Magnesium perchlorate, 8-20 mesh.
   8.2 Argon, 99.9 percent purity.
   8.3 Oxygen, 99.9 percent purity.
   8.4 Tetrahydrofuran, 99 percent Nitrogen free.
   8.5 Charcoal, activated 8-20 mesh.

2.15.2.3 Procedure.

Assemble the pyro-reactor and nitrogen detector and follow all operating instructions per the Antek Operation Manual. Set the following operating parameters:

Oxygen Flow: 120 ml/min. to Ozone Generator
              410 ml/min. to Pyrolysis Tube

Argon Flow: 30 ml/min.

Pyro-Reactor Temperature:

          Inlet - 850 degrees Celsius
          Center - 950 degrees Celsius
          Outlet - 950 degrees Celsius

Instrument

Sensitivity: I-Low (counts between 300 and 60,000)

Injection

Rate: 1.0 = 1/second

Into a tared 10 ml vial, dispense 0.8 ml of AMK and 3.2 ml of tetrahydrofuran (THF). Weigh each component to the nearest 0.1 mg. Seal the vial tightly with a solvent resistant cap and mix thoroughly by shaking. Let the sample stand long enough to dissipate the bubles. Then inject 3 consecutive (3.0 µl) samples of AMK into the nitrogen analyzer using the microsyringe attached to the constant

rate syringe drive. Record the number of counts displayed on the digital readout of the nitrogen detector after each sample injection. The average number of counts from these three injections can be used to calculate the nitrogen content of the AMK sample from a calibration curve similar to the one shown in figure 2.38. Because the density of the diluted AMK samples is different than the standards, a density correction factor (density of standards/density of diluted AMK solutions) is applied to the average AMK counts. For practical purposes, the density of the AMK samples is essentially that of THF (0.889 g/ml). Consequently, a correction factor of 0.975 would account for the slightly larger mass of AMK injected. Finally, in order to convert the nitrogen content of the AMK sample to parts per million (ppm) amine the dilution of the sample and the weight fraction nitrogen in the amine must be accounted for. A sample calculation is shown below.

1. Average counts for diluted AMK (uncorrected for density) = 1000.

2. Average counts for diluted AMK (corrected for density) = 1000 X 0.975 = 975.

3. PPM nitrogen in diluted AMK (from figure 2.38) = 5.8.

4. PPM nitrogen in undiluted AMK (from dilution factor) = 5.8 (wt fraction AMK in THF) = $\frac{5.81}{0.21}$ = 27.6

5. Wt% nitrogen in amine = $\frac{14.0 \text{ X } 100}{\text{Amine Mol. Wr.}}$

6. PPM amine in AMK = $\frac{27.6}{\text{Wt \% Nitrogen}}$ = 100 = 1.97 X Amine Mol. Wt.

NOTE: A new calibration curve should be established daily or anytime after the instrument has been shut down.

In order to establish a calibration curve, it is recommended that at least three consecutive (3.0 μl) injections should be made for each standard. Furthermore, at least three dilutions should be used to produce counts in the same range as AMK. For the highest precision, the calibration curve should be checked before and after anlaysis of AMK samples.

2.15.2.4 RESULTS AND DISCUSSION.

The amine content of antimisting fuel can be determined by combining combustion with nitrogen detection by chemiluminescence. The relative sensitivity of this test method is a few parts per billion of nitrogen. The results of some initial testing done by SWRI are shown below; the results show the mean and standard deviation (based on five consecutive injections) for 3 FM-9 AMK blends.

	RMH1-129	RMH1-136	RMH1-138
X =	181 ppm	112 ppm	114 ppm
=	5 ppm	6 ppm	11 ppm
* =	150 ppm	135 ppm	150 ppm

* The expected amine content, based on the amine content being a specified percent of the nominal polymer content of the AMK as indicated on the sample containers.

The difference between the measured and the expected amine content of these samples could be due to the fact that the nominal polymer content may be slightly different than the actual polymer content of the samples.

2.16 DENSITY DETERMINATION.

2.16.1 Background and Theory.

The determination of density for antimisting fuel is a straight forward measurement of mass per unit volume. The measurement of mass and volume is done at a specified temperature of 25°C.

The density measurement, while having its own importance, is also used in determining the dynamic viscosity of an AMK sample. The method for determining the dynamic viscosity is given in the Viscosity Ratio Test Section of this report and follows this general formula:

Dynamic Viscosity (cps) = Density (g/ml) x Kinematic Viscosity (cSt)

2.16.2. Experimental.

2.16.2.1 Apparatus.

The apparatus used to determine the density of an AMK sample consists of common laboratory equipment available in most chemical laboratories. A measured amount of antimisting fuel is placed in a graduate cylinder of known weight. The graduate cylinder is placed in a constant temperature water bath where the AMK sample is allowed to equilibrate to the bath temperature. After the graduate cylinder and the AMK sample reach the specified test temperature, the cylinder is removed and immediately weighed. A reading is also made of the volume of the AMK sample in the graduate cylinder. The density can then be determined by dividing the AMK sample weight by the AMK sample volume. The density is then reported in mg per ml.

2.16.2.2 Equipment Specifications.

1. Graduate cylinder, 25 ml, 0.2 ml divisions.

2. Balance, capable of weighing to 0.1 mg.

3. Constant temperature water bath, capable of maintaining a temperature +0.05°C.

4. Aluminum foil.

5. Thermometer, of applicable range.

2.16.2.3 Procedure.

1. Record the tare weight (nearest 0.001 gm) of a 25 ml graduated cylinder.

2. Add approximately 23 mls of sample to the graduate. Pour slowly to avoid splashing the sample on the upper portion of the graduate.

3. Record the weight of the graduate plus sample.

4. Put a piece of aluminum foil over the top of the graduate and place the graduate in the 25°C constant temperature bath. The level of water in the bath should be slightly above the level of sample in the graduate.

5. Allow the sample to come to temperature.

6. Remove the graduate from the bath, observe the bottom of the sample meniscus and record this volume to the nearest 0.05 ml.

7. Calculate the density:

$$\text{Density} = \frac{(\text{wt sample} + \text{wt graduate}) - \text{wt graduate}}{\text{mls sample}} = \text{mg/ml}$$

2.16.2.4 Results and Discussion.

The procedure for determining the density of an antimisting fuel sample is not complex. Accurate and repeatable results can be obtained through careful weighing, volume measurement, and temperature control during the test procedure for density determination.

2.17 PARTICLE SIZE EVALUATION FOR FM-9 SLURRY.

2.17.1 Background and Theory.

During the early development of the inline blending process for antimisting fuel, there were problems related to the inline blending hardware and procedures as well as problems related to the FM-9 slurry. FM-9 polymer itself is a dry, granular substance. In order to allow FM-9 polymer to be pumped and metered, the polymer was mixed with a carrier fluid which became known as FM-9 slurry. This slurry could be pumped relatively easily using a "progressive cavity" rotary screw pump and it could be metered accurately by varying the speed of the pump.

As antimisting fuel began to be produced using these inline blenders and slurry it became apparent that some of the AMK blends being produced contained "fish eyes" that tended to settle on the bottom of the fuel container and form a layer of thick gel. A "fish eye" is a large, partially dissolved, FM-9 polymer particle that is surrounded by a layer of "clear gel" containing a high concentration of dissolved polymer. These "fish eyes" seemed to be caused by large FM-9 polymer particles not being able to rapidly disperse and dissolve during the blending process. This settling process resulted in AMK with a low polymer concentration with reduced flammability protection as well as compatibility problems in fuel systems.

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Continuing research revealed that these "fish eyes" could effectively be reduced in numbers or alleviated altogether if the FM-9 slurry was sieved to remove any particles of FM-9 polymer above a certain size before the slurry was used in the inline blenders to produce AMK. JPL developed this test method to determine the presence of large FM-9 polymer particles in FM-9 slurry as it was received from ICI. The use of this test method lead JPL to establish criteria for an acceptable quality of FM-9 slurry relative to FM-9 polymer particle size.

2.17.2 Experimental

2.17.2.1 Apparatus

The particle size evaluation test apparatus is made up of off-the-shelf laboratory equipment which is commonly available in most laboratories. The contents of a beaker that contains a measured amount of FM-9 slurry which is diluted with water is poured through a sieve of known mesh size. The resulting residue on the sieve is then dried and weighed. The residue is then compared to the original slurry weight on a weight-to-weight basis.

2.17.2.2 Equipment Specifications.

1. "Tyler" sieve, 200 mesh.

2. "Griffin" beaker, 2000 ml.

3. Magnetic stirrer.

4. Automatic balance, capable of reproducing weights to 0.1 mg.

5. Methyl alcohol.

6. Water, tap.

7. Drying oven.

8. Hobart mixer.

2.17.2.3 Procedure.

1. Thoroughly mix a container (pail) of slurry as received from ICI, using a Hobart mixer or other suitable means.

2. Weigh out 100 grams of slurry and place the slurry in a 2000 ml graduated "Griffin" beaker that has a magnetic stirrer bar in it.

3. Set the stirrer on low.

4. Slowly, dilute the slurry with 1500 ml of tap water.

5. Continue stirring until the liquid is homogeneous and has the consistency of milk.

6.   Pour the contents of the beaker through a 200 mesh "Tyler" sieve.

7.   Wash the material which remains on top (if any) of the sieve first with water and then with methyl alcohol.

8.   Set the drying oven at 50°C.

9.   Place the sieve in the oven and dry to a constant weight.

10.   Allow the sieve to cool. Collect and weigh the residue on the sieve. Record the weight.

11.   Calculation:

$$\frac{\text{weight of residue powder/particles} \times 100}{\text{weight of slurry}} = \% \text{ particles}$$

2.17.2.4   Results and Discussion.

This test method that JPL developed can detect the presence of large FM-9 polymer particle agglomerates in FM-9 slurry that can cause blending problems such as polymer settling and gel formation (reference 13). Research done by using sieves of various mesh sizes determined that the above blending problems could be overcome if FM-9 polymer particles over the 100 micron size were removed from the slurry before blending. A criteria for an acceptable quality FM-9 slurry was established by using a 200 mesh "Tyler" sieve. A 200 mesh "Tyler" sieve allows particles of 75 microns or less to pass through the sieve. FM-9 slurry with less than 0.05 percent of particles above the 75 micron size is considered to be an acceptable quality slurry, and it is practically free of particles above the 100 micron size (reference 13). ICI has since alleviated this problem by maintaining a closer tolerance on the FM-9 particle size during the production stages of the slurry. Since then, the particle size evaluation has been used to spot check slurry produced from different polymer lots, and there is no longer a need for screening the slurry before use in the inline blenders.

2.18   SLURRY VISCOSITY DETERMINATION.

2.18.1   Background and Theory.

Early in the antimisting fuel program it became apparent that for the use of antimisting fuel to be feasible it would be necessary to inline blend the FM-9 additive with the Jet A at the aircraft's refueling point. In April 1983, the first inline blended fuel was used to fuel the SP-2H aircraft at the Lakehurst Naval Air Station for a large-scale aircraft crash test. Before this time, all of the antimisting fuel used for testing was produced by batch blending at the ICI plant in Wilmington, Delaware.

The first inline blended AMK was blended using FM-9 slurry. The slurry consisted of FM-9 polymer mixed with an isopropyl amine which served as a carrier fluid. During the development and early use of several different types of FM-9 slurry, the consistency and viscosities of the slurries varied greatly. The consistencies of these slurries varied from a water-like substance to a solid-like substance. The viscosities varied during the slurry development process because it was trying to optimize the polymer loading and the blendability of the slurry.

The slurry had to meet two requirements in order for it to be used in an inline AMK blender. First, the slurry had to have a viscosity that allowed the material to be pumped; and second, the slurry had to be able to be metered after it was pumped. Besides these blender requirements, the slurry had to exhibit good dispersion characteristics in Jet A, rapid equilibration, and yield adequate fire protection in the resulting antimisting fuel.

After an acceptable slurry began to be produced on a large-scale basis, it became apparent that there were batch to batch variations in the viscosity of the slurries. It, therefore, became important to assess the variations in the viscosity of the slurry and specify a standard for the viscosity of FM-9 slurry. The device used to measure the viscosity of the slurry is a Brookfield Synchro-Lectric Viscometer fitted with a special T-bar type spindle used with a Brookfield Helipath stand. The complete apparatus is shown schematically in figure 2.39. This apparatus will permit viscosity measurements in relative centipoise values of materials having characteristics similar to past, putty, or cream.

2.18.2 Experimental.

2.18.2.1 Apparatus.

The Brookfield Syncnro-Lectric Viscometer operates by rotating a cylinder, a disc or a T-bar spindle in a fluid, or a substance and measuring the torque necessary to overcome the viscous resistance to the induced movement. This measurement is accomplished by driving the immersed spindle through a calibrated spiral spring to the degree to which the spring is wound, indicated by the position of the pointer on the dial of the viscometer as shown in figure 2.40. The pointer reading is proportional to the viscosity of the fluid or substance for any given rotation speed and spindle size. The viscometer is able to measure over a number of ranges since, for a given drag, or spring deflection, the actual viscosity is proportional to the spindle speed and is also related to the spindle's size and shape. For a material of a given viscosity, the drag will be greater as the spindle size and/or rotational speed increase.

The measurement of the materials with normal flow properties is based on a reversible isothermal change in apparent viscosity with change in shear rate (reference 22). The measurement is performed using the Brookfield Viscometer under standardized conditions with rigid control of the time intervals of measurement. Readings are obtained on the viscometer dial scale at the end of 1 minute for each rotational speed. Changes from the lowest speed to the highest speed, and return to the lowest speed, are made without stopping the instrument. The thixotropic index is determined from the ratio of the apparent viscosity at the second 6 RPM speed over the apparent viscosity at the 60 RPM speed.

A rotating member, such as a cylinder or a disc, when placed in a semi-solid substance such as FM-9 slurry will create a channel in the substance and a meaningless torque will be exerted on the spindle of the viscometer. Also, the viscosity of the slurry will tend to decrease when subjected to the internal shearing created by a rotating member in the slurry and the torque required to rotate the spindle will decrease as the period of shearing increases. For these reasons, FM-9 slurry is not suitable for viscosity measurements using the Brookfield Viscometer with cylinder or disc spindle as normally used for materials with flow properties. Instead, the measurement of a material without
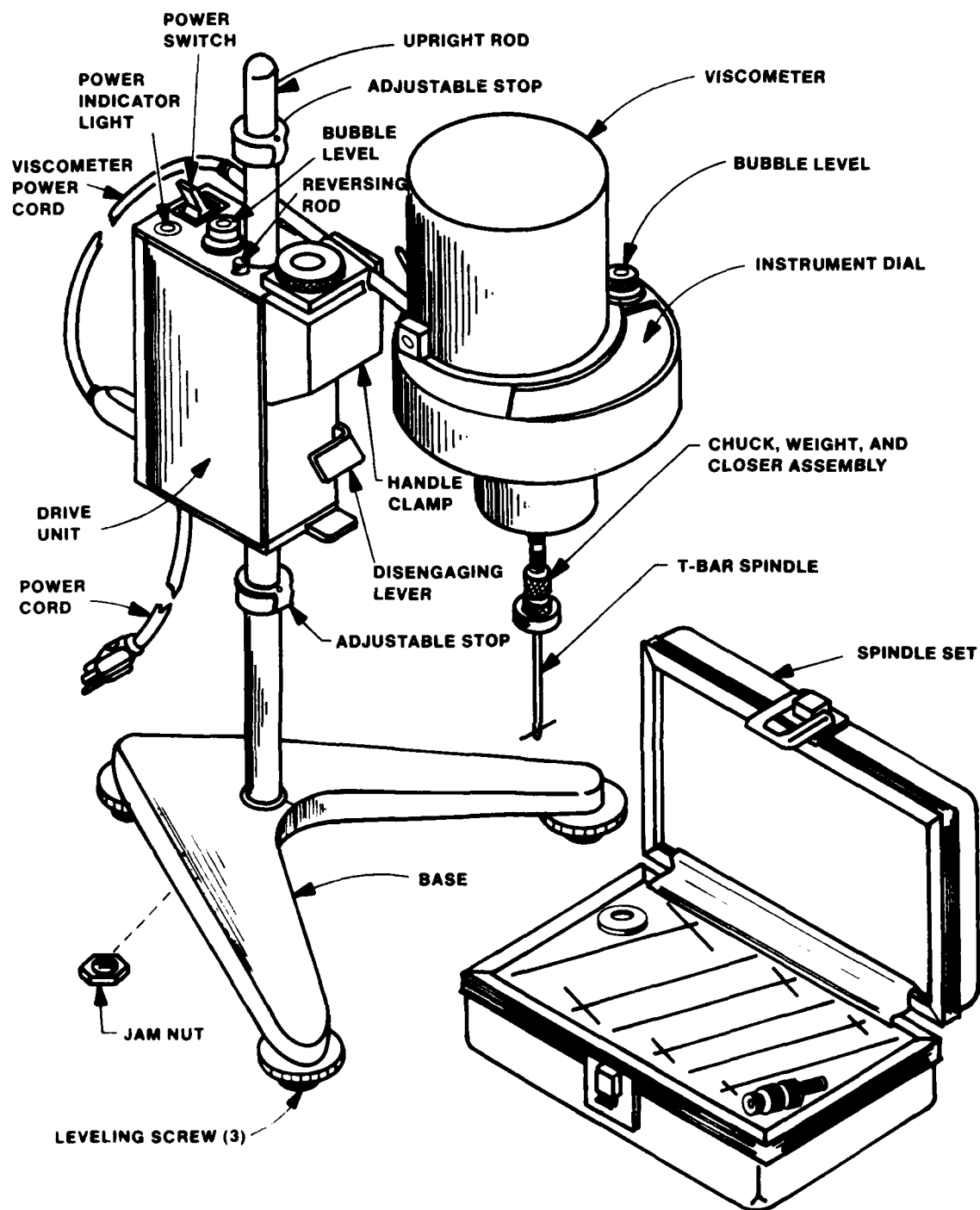
FIGURE 2.39 SCHEMATIC OF BROOKFIELD VISCOMETER, HELIPATH STAND, AND T-BAR SPINDLES

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Labels on the left (top to bottom): SYNCHRONOUS MOTOR, SPEED SELECTOR KNOB, ON-OFF TOGGLE SWITCH, CLUTCH LEVER, KNURLED NUT, HANDLE, POINTER, JEWEL BEARING SUPPORT, SPINDLE COUPLING NUT, IMMERSION MARK, SPINDLE BODY

Labels on the right (top to bottom): GEAR TRAIN, CIRCULAR BUBBLE LEVEL, DIAL, CALIBRATED SPIRAL SPRING, UPPER SHAFT, PIVOT POINT, REMOVABLE SPINDLE GUARD, SAMPLE CONTAINER

FIGURE 2.40   SCHEMATIC OF BROOKFIELD VISCOMETER'S
INTERNAL MECHANISM

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flow properties is performed with a spindle in the form of an inverted T attached to the Brookfield Viscometer which is supported by a Helipatn Stand (reference 22). As the spindle is rotated by the viscometer, the delipath Stand lowers it through the teat material. Throughout its helical path the crosspiece constantly encounters new material; thus, the rotor always measures material which has an undisturbed structure. The torque required to rotate the spindle is registered on the viscometer dial and is converted to centipoise values.

2.18.2.2 Equipment Specifications.

1. Brookfield Viscometer, Syncnro-Lectric, Model LVT or HBT, manufactured by Brookfield Engineering Laboratories, Stoughton, Massachusetts (see figure 2.41).

2. Brookfield Helipath Stand.

3. Brookfield T-Bar Spindle set with chuck.

4. Thermometer, precision, capable of indicating $25^{\circ}C$ with graduations not greater than $0.2^{\circ}C$ (0.5° F).

5. Constant temperature bath capable of maintaining 25° $\pm 0.5^{\circ}C$ ($\pm 1^{\circ}$ F).

6. Jars, 16-ounce wide mouth, 3-inch minimum diameter.

2.18.2.3 Procedure.

1. Fill a 16-ounce jar to bottom threads with the material to be tested and mix thoroughly using a spatula.

2. Immerse closed jar in bath and allow the jar to come to a temperature of $25^{\circ}C$ $\pm 0.5^{\circ}C$. Check temperature of sample using a precision thermometer.

3. Follow all instructions and precautions stated in the instruction manual that is included with each Brookfield Viscometer.

4. Refer to the attached Helipath Stand Spindle Ranges sheet and select a T-bar spindle. (NOTE: Spindles should be chosen so that the initial reading is about 50 percent of full-scale deflection at the lowest possible speed.)

5. Slide the spindle into the closer assembly and tighten.

6. Level the viscometer and set the spindle so that the crosspiece is covered by about 1/4-inch of test material.

7. Set the adjustable stops for the spindle penetration required according to the depth of the sample. (NOTE: Push the reversing rod down for initial drive direction.)

8. Adjust the hexagonal knob on the viscometer to the lowest speed possible for optimum readings at about 50 percent of full-scale deflection. (NOTE: The highest speed should not exceed 5 RPM for the HBT Model Viscometer).
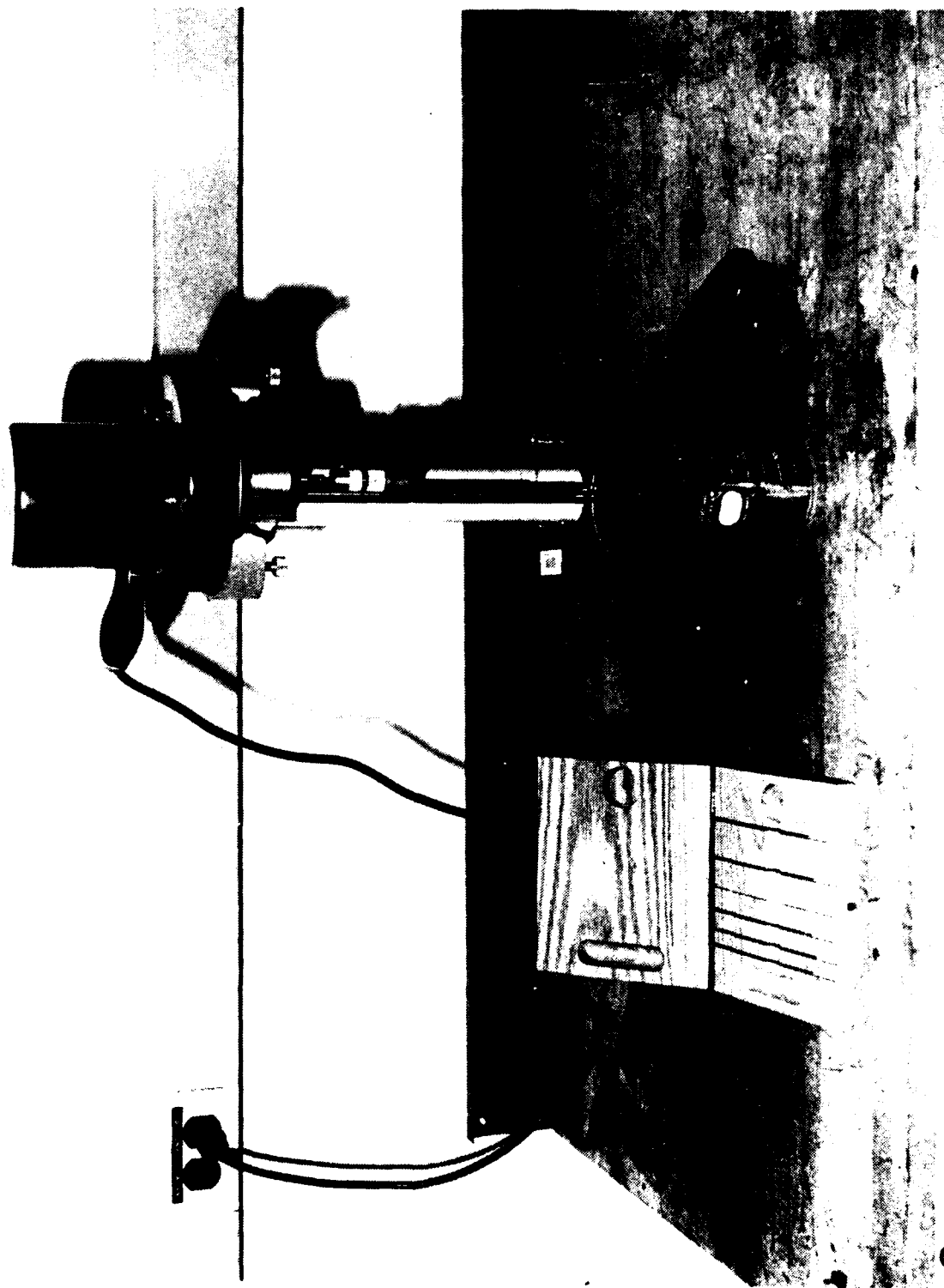
FIGURE 2.41 BROOKFIELD VISCOMETER

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9. Start the viscometer and let the dial make one or two revolutions before turning on the Helipath Stand.

10. Turn on Helipath Stand and record all dial re#dings as the needle passes a predetermined spot in the viewing glass.

11. Determine and report the viscosity of the highest and lowest readings as well as the average of all the readings.

12. Calculation:

Apparent Viscosity (cps) = actual scale reading X (0.01) X the factor for the RPM and spindle used (see table 2.3).

Sample calculation and report shown in table 2.4.

TABLE 2.3 HELIPATH STAND RANGE DATA FOR T-BAR SPINDLES

MODEL LV AND HA VISCOMETERS

SPINDLES	T-A	T-B	T-C	T-D	T-E	T-F
LVT 0.3 RPM	66.6M	133M	333M	666M	1.66MM	3 33MM
LVT 0.6 RPM	33.3M	66.6M	166M	333M	833M	1.66MM
LVT 1.5 RPM	13.3M	26.6M	66.6M	133M	333M	666M
LVT 3 RPM	6.66M	13.3M	33.3M	66.6M	166M	333M
LVT 6 RPM LVF 6 RPM	3.33M	6.66M	16.6M	33.3M	83 3M	166M
LVT 12 RPM LVF 12 RPM	1.66M	3.33M	8.33M	16.6M	41.6M	83.3M
HAT 0.5 RPM	800M	1.6MM	4MM	8MM	20MM	40MM
HAT 1 RPM HAF 1 RPM	400M	800M	2MM	4MM	10MM	20MM
HAF 2 RPM	200M	400M	1MM	2MM	5MM	10MM
HAT 2.5 RPM	160M	320M	800M	1.6MM	4MM	8MM
HAT 5 RPM HAF 5 RPM	80M	160M	400M	800M	2MM	4MM

M = 1,000   MM = 1,000,000

MODEL RV AND HB VISCOMETERS

SPINDLES	T-A	T-B	T-C	T-D	T-E	T-F
RVT 0.5 RPM	400M	800M	2MM	4MM	10MM	20MM
RVT 1.0 RPM	200M	400M	1MM	2MM	5MM	10MM
RVF 2.0 RPM	100M	200M	500M	1MM	2.5MM	5MM
RVT 2.5 RPM	80M	160M	400M	800M	2MM	4MM
RVF 4 RPM	50M	100M	250M	500M	1.25MM	2.5MM
RVT 5 RPM	40M	80M	200M	400M	1MM	2MM
HBT 0.5 RPM	3.2MM	6.4MM	16MM	32MM	80MM	160MM
HBT 1 RPM HBF 1 RPM	1.6MM	3.2MM	8MM	16MM	40MM	80MM
HBF 2 RPM	800M	1.6MM	4MM	8MM	20MM	40MM
HBT 2.5 RPM	640M	1.28MM	3.2MM	6.4MM	16MM	32MM
HBT 5 RPM HBF 5 RPM	320M	640M	1 6MM	3.2MM	8MM	16MM

M   1,000   MM   1,000,000

TABLE 2.4 CALCULATION AND REPORT EXAMPLE

ANALYST   HJBJ   DATE   2/15/83

   SAMPLE ID   JCK 11-66

   MODEL NO. OF INSTRUMENT   HBT 73121

   TEMPERATURE OF SAMPLE   25°C

SPINDLE #	RPM	FACTOR*	SCALE READING	APPARENT VISCOSITY (cps)
T-E	1	(0.01) (3.2mm)	33	1,056,000
			35	1,120,000
			35	1,120,000
			34	1,088,000
			37	1,184,000
			36	1,152,000
			35	1,120,000
			37	1,184,000
			40[1]	1,280,000
			37	1,184,000
			35	1,120,000
			35	1,120,000
			36	1,152,000
			34	1,088,000
			31[2]	992,000
			32	1,024,000
			AVG. 35[3]	1,120,000

[1]Highest Apparent Viscosity (cps)=40 X(0.01)X (3,200,000)=1,280,000
[2]Lowest Apparent Viscosity (cps)=31 X(0.01)X (3,200,000)=992,000
[3]Average Apparent Viscpsity (cps)=35 X(0.01)X (3,200,000)=1,120,000
*m= 1,000
mm= 1,000,000

2.18.2.4 Results and Discussion.

The Brookfield Synchro-Lectric Viscometer fitted with a T-bar type spindle and used with a Brookfield Helipath Stand will produce accurate viscosity measurements in relative centipoise values for FM-9 slurry. The specification for the viscosity of standard FM-9 slurry is 35,000 centipoise (maximum) at 68 degrees Fahrenheit. The viscosity specification was jointly established by FAA and ICI by placing the following conditions on the slurry:

1. Compatibility with blending hardware (i.e., pumpability, meterability).

2. Adequate dispersion (blendability).

3. Rapid equilibration.

4. Adequate fire protection of the resulting AMK.

The slurry can have a viscosity lower than 35,000 centipoise if it still meets the above requirements and for blending operations the lower the slurry viscosity is, the easier it is to pump and meter. A typical standard FM-9 slurry exhibits a viscosity value of 13,000 centipoise.

During the course of the AMK program, a standard test procedure for slurry viscosity determinations relative to spindle size and RPM was adopted by the FAA and ICI. The procedure specifies a T-A T-bar spindle be used at a viscometer speed setting of 5 RPM to report the average viscosity of the slurry sample. This allowed researchers that were making viscosity measurements of FM-9 slurry to report values that should be comparable and aided in the interpretation of viscosity values of slurries from different batches and of different types.

3. GENERAL FUEL SAMPLE INFORMATION.

1. All laboratory characterization tests, unless otherwise specified in the test procedure, are to be performed with the test fuel sample at a temperature of 20 degrees ±2 degrees Celsius.

2. The rapid heating or cooling of the test fuel sample to obtain the specified test fuel temperature must be avoided because it can affect the results of the laboratory characterization tests.

3. All laboratory characterization tests should be performed in a clean and draft-free environment with an ambient temperature between 20 degrees Celsius and 25 degrees Celsius.

4. The contact of the test fuel sample with water (liquid or vapor) must be avoided because it can affect the results of the laboratory characterization tests. The test fuel sample should be discarded if there is a known contact of the sample with liquid water.

5. The recommended cleaning solvent for all the laboratory characterization test equipment and glassware is 1,1,1-trichloroethane.

6. The recommended sample container is a 1-gallon rectangular tin-plated steel can with a knurled air tight cap as per Federal Specification PPP-C-96 (common 1-gallon oblong cans used for products such as paint thinner). Sample containers should not be reused.

7. The exposure of antimisting fuel to light should be avoided because light can affect the antimisting properties of the AMK.

4. GENERAL SAFETY PRECAUTIONS.

1. It is recommended that all laboratory characterization tests and all cleaning of the test equipment be performed under a fume hood or an area with adequate draft-free ventilation.

2. A respirator with proper cartridges should be worn when handling FM-9 slurry, Jet A fuel, and antimisting fuel to avoid inhaling the fumes.

3. Safety glasses, rubber gloves, and protective clothing should be worn when handling FM-9 slurry, Jet A fuel, and antimisting fuel to avoid contact with the skin or eyes.

4. All general laboratory safety precautions should be followed as well as following any specific safety precautions relative to handling kerosene, 1,1,1 trichloroethane, amine, glycol, and sodium hydroxide.

5. ICI Americas Inc. Material Safety Data Sheet (001438), which is shown in appendix D, should be referred to for the proper and safe handling of Avgard slurry.

5. CONCLUSIONS.

1. Laboratory characterization tests are available to adequately evaluate antimisting fuel relative to the following:

    a. The antimisting properties of batch and inline blended AMK.

    b. The degree of intentional degradation produced by a degrader.

    c. The degree of unintentional degradation produced in an aircraft fuel system.

    d. The filtration characteristics of degraded AMK.

    e. The composition and physical properties of AMK and FM-9 slurry.

2. The primary quality control tests for antimisting fuel, available at the time of the Controlled Impact Demonstration (December 1984), were the Filter Ratio Test, the Cup Test, the Solids Test, and the Turbidity (Clarity) Test. These laboratory characterization tests were initially developed to characterize FM-9 antimisting fuel in the beginning of the antimisting fuel program, and the tests have remained the primary quality control tests throughout the AMK program.

3. One of the primary goals throughout the antimisting fuel program was to develop a single quality control test that could be used to determine if the antimisting fuel that was inline blended into an aircraft was acceptable quality AMK before the aircraft proceeded for takeoff. Much work was done to develop real-time quality control tests for antimisting fuel. The only standardized laboratory characterization test that was developed as a near real-time quality control test was the Die Swell Test.

4. The laboratory characterization tests that are included in this report, and the specifications that were established for FM-9 slurry, antimisting fuel, and degrader performance, should provide the necessary information to assess the quality of the fuel relative to fire protection and aircraft system compatibility. The specifications are shown in appendices A, B, and C.

5. The normal stress and extensional viscosity properties of FM-9 antimisting fuel are considered to account for the antimisting performance of the fuel. Attempts to make direct measurements of the normal stress and extensional viscosity properties of FM-9 antimisting fuel at high shear rates have been unsuccessful to date.

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13. [illegible]

APPENDIX A
FM-9 SLURRY SPECIFICATION

1. Slurry Constituency

Solids = 25.0 ± 0.5 percent of total weight.
Amine* - 1.25 ± 0.25 percent of total weight.
Water = 1.0 ± 0.5 percent of total weight.
Glycol* = remainder, or about 73 percent of total weight.

2. Slurry viscosity = 35,000 centipose (maximum) at 68 degrees Fahrenheit.

3. Slurry exposure to light must be controlled.

4. Slurry shelf life (no permanent separation of constituents) = 2 months
(minimum). Mild agitation of slurry during blending to control temporary
constituent separation is acceptable.

The only restriction seen necessary to standard (1983) ASTM D-1655 Jet A kerosene
specification is that the aromatic content of the base Jet A must be at least 12
percent by volume to assure quality AMK.

*NOTE: The specific glycol and amine types remain proprietary to the
manufacturer, ICI Americas Inc.

APPENDIX B
FM-9 ANTIMISTING FUEL SPECIFICATION FOR THE
CONTROLLED IMPACT DEMONSTRATION

1. The AMK fuel shall have 1.2 percent $\pm$ 0.04 percent by weight of slurry
(25 percent polymer loading) which is controlled during blending process to
achieve an FM-9 polymer/Jet A fuel concentration = .30 percent as verified by the
solids test (ASTM D381, existent gum, wt/wt). NOTE: If the blended fuel does
not meet these specifications, it must be discarded.

2. After a minimum of 12 hours after completion of blending operation, the
characterization test results must meet the following specifications:

> Clarity (NTU) = 20 max
> Orifice Flow Cup (ml/30 secs) = 1.7 min to 2.3 max
> Filter Ratio = 35 min to 90 max
> Total Water (ppm) (ASTM D1744-64) = 230 max
> Uniformity = No visible or residual sediment in 5 gallons of
> blended AMK fuel.

APPENDIX C
ACCEPTABLE DEGRADER PERFORMANCE SPECIFICATION FOR THE
CONTROLLED IMPACT DEMONSTRATION

The CID aircraft degrader shall produce degraded AMK with the following
specifications (samples taken at engine fuel pump inlet while the engine is
running):

1. Filter Ratio = 1.2 max
2. Orifice Flow Cup = 7.0 min
3. Transition Velocity = 2 to 3 cm/sec

MATERIAL SAFETY DATA SHEET

ICI Americas Inc.

Wilmington, Delaware 19897

Phone (302) 575-3000 (24 hours)

001483

Form No.: 3524h
Date: 2/84

SECTION 1 NAME & PRODUCT

Material name:
Avgard Additive SD (25%)

SECTION 2 INGREDIENTS

	%	TLV (ACGIH)
Polymer	25	Not listed
Proprietary Solvents	75	See Section 8

Not specification values. qt = greater than, lt = less than, ca = approximately

SECTION 3 PHYSICAL DATA

Boiling point: Above 350°F (estimated)
Vapor pressure (mmHg at 20°C): Below 0.1 (estimated)
Vapor density (air = 1): Not applicable
Solubility in water: Partially
pH: 9.0
Specific gravity: 1.09
% Volatile by volume: No data
Appearance and odor: White paste with amine odor

SECTION 4 FIRE AND EXPLOSION HAZARD DATA

Flash point (and method): Not applicable
Autoignition temp.: No data
Flammable limits (STP): No data
Description: Combustible solid

Extinguishing media:
Water fog, alcohol foam, carbon dioxide, dry chemical, halon 1211. Water or foam
may cause frothing.

Special fire fighting protective equipment:
Self-contained breathing apparatus with full facepiece and protective clothing.

Unusual fire and explosion hazards:
None known.

SECTION 5 REACTIVITY DATA

Stability:
Stable under normal conditions.

Incompatibility (materials to avoid):
Oxidizing agents.

Hazardous decomposition products:
Combustion products: carbon dioxide, carbon monoxide, nitrogen oxides.

SECTION 5 REACTIVITY DATA (continued)

Hazardous polymerization:
   Will not occur.

SECTION 6 HEALTH HAZARD ASSESSMENT

General:
   Limited toxicity data are available on this specific product. The health hazard
   assessment is based on the results of screening data.

Ingestion:
   The acute oral LD_{50} in rat is above 5.0 g/kg. Relative to other materials, a
   single dose of this product is slightly toxic by ingestion, Hodge, H.C. and
   Sterner, J.H., American Industrial Hygiene Association Quarterly, 10:4, 93,
   Dec. 1949.

Eye contact:
   This material is severely irritating in rabbit eye studies and similar degree of
   irritation will probably occur after human eye contact.

Skin contact:
   This material is nonirritating in rabbit dermal irritation studies and no
   irritation is expected to develop after contact with human skin. It was not a skin
   sensitizer in animal testing. However, irritation could develop from repeated
   and/or prolonged skin contact.

Skin absorption:
   This product is probably not absorbed through human skin.

Inhalation:
   Vapors and aerosols can produce irritation of eyes, nose and throat.

Effects of overexposure:
   Vapors and aerosol forms are irritants.

First aid procedures:
   Skin: Wash material off the skin with copious amounts of soap and water. If
   redness, itching or a burning sensation develops, get medical attention.
   Eyes: Immediately flush with copious amounts of water for at least 15 minutes and
   have eyes examined and treated by medical personnel.
   Ingestion: Give one or two glasses of water to drink. If gastrointestinal
   symptoms develop, consult medical personnel. (Never give anything by mouth to an
   unconscious person.)
   Inhalation: Remove victim to fresh air. If cough or other respiratory symptoms
   develop, consult medical personnel.

SECTION 7 SPILL OR LEAK PROCEDURES

Steps to be taken in case material is released or spilled:
   Wear skin, eye and respiratory protection during cleanup. Shovel up and recover or
   mix material with absorbent and shovel into waste container. Cover container and
   remove from work area. Wash down spill area with hot water containing detergent
   and flush away with plenty of water to sanitary drain or sewer serviced by a
   wastewater treatment facility.

SECTION 7 SPILL OR LEAK PROCEDURES (continued)

Disposal method:
  Dispose of waste in facility permitted for nonhazardous waste.

Container disposal:
  Do not distribute, make available, furnish or reuse empty container except for
  storage and shipment of original product. Empty container of all residue.
  Puncture or otherwise destroy empty container and dispose of in facility permitted
  for nonhazardous waste.

SECTION 8 SPECIAL PROTECTION INFORMATION

TLV® or suggested control value:
  No value assigned to this mixture. Minimize exposures in accordance with good
  hygiene practice. Proprietary solvents do have ACGIH TLV® values.

Ventilation:
  Use local exhaust to control exposures.

Respiratory protection (specify type):
  If needed, use MSHA/NIOSH approved respirator for dusts, mists and fumes whose TLV
  is greater than 0.05 mg/m^3 in combination with a cartridge for ammonia and methyl
  amines.

Protective clothing:
  Impervious gloves, apron and arm covers. Additional protection may be required
  depending upon conditions of use.

Eye protection:
  Chemical tight goggles with full faceshield.

Other protective equipment:
  Eyewash station and safety shower in work area.

SECTION 9 SPECIAL PRECAUTIONS OR OTHER COMMENTS

Precautions to be taken in handling or storing:
  Avoid skin and eye contact.

<div align="center">
The information herein is given in good faith
but no warranty, expressed or implied, is made.
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APPENDIX E

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ADL-32 North	(1)	APA-300	(1)	ALG-300	(1)
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ANM-50	(2)				

E-1

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